



Effect of Various Cleaning Methods on Microshear Bond Strength of Contaminated Yttria-Stabilized Tetragonal Zirconia Polycrystal Ceramic to Self-Adhesive Resin Cement, and Surface Wettability

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ABSTRACT

Objectives: This study evaluated the impact of different cleaning methods on microshear bond strength (μ SBS) of contaminated yttria-stabilized tetragonal zirconia polycrystal ceramic to self-adhesive resin cement, and surface wettability.

Materials and Methods: In this in vitro study, sandblasted zirconia plates measuring $10 \times 10 \times 3$ mm were randomly divided into 6 groups: a non-contaminated (NC) negative control group and 5 experimental groups that were contaminated with human blood and saliva, and cleaned with (c) air-water spray (CW) as positive control, (c) 70% ethanol (CE), (d) 1.0wt% sodium hypochlorite (SHC), (e) Zirclean (CZ), and (f) nonthermal atmospheric plasma (NTAP) (air plasma type). Surface wettability was then calculated. Sixteen Tygon tubes (0.8×2 mm) filled with Theracem self-adhesive resin cement were bonded to the zirconia specimens, and underwent μ SBS testing after one week of storage. Data were analyzed by one-way ANOVA and Tukey's test ($\alpha=0.05$).

Results: SHC and NTAP yielded the highest μ SBS values, with no significant difference with the NC group ($P>0.05$). CW produced the lowest μ SBS, which was not significantly different from the CE and CZ ($P>0.05$). CE showed the highest contact angle, which was not significantly different from the NC and CZ ($P>0.05$); SHC produced the lowest contact angle. There was no significant difference in contact angle between NTAP and CW ($P>0.05$), but their contact angle was larger than that of SHC ($P=0.014$).

Conclusion: Cleaning zirconia with SHC and NTAP controlled the impact of saliva and blood contamination, and produced a μ SBS to self-adhesive resin cement comparable to the positive control.

Keywords: Decontamination; Plasma Gases; Shear Strength; Yttria-Stabilized Tetragonal Zirconia

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INTRODUCTION

The partially stabilized zirconia utilized in dentistry possesses high flexural strength, high flexural modulus, and optimal biocompatibility [1]. Zirconia ceramics possess great mechanical characteristics;

however, the resin cement-zirconia bonding is dependent on surface pretreatment. The application of conventional hydrofluoric acid along with silanization, which has been proven to be an efficient surface conditioning and treatment technique for silica-based ceramics

[2], is not suitable for zirconia. Since zirconia lacks a glass phase, hydrofluoric acid cannot etch the zirconia surface [3]. An appropriate method to obtain a durable bond to zirconia has not been fully clarified. Hence, substitute surface conditioning techniques such as physicochemical activation of the ceramic surface with silica-coated alumina particles and chemical activation with primers or resin cements containing phosphate monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) are gaining popularity [2]. Resin cements typically necessitate the pretreatment of both tooth and ceramic surfaces, utilizing a range of materials across distinct phases. In order to streamline this process, self-adhesive cements were developed [3]. The *in vitro* adhesion efficacy of self-adhesive cements to diverse restorative materials is comparable to that of traditional multistep resin cements [3]. Nevertheless, the existing literature regarding their clinical efficacy remains inadequate [3].

A review of literature [4] showed the best clinical proof available for effective bonding of dental oxide ceramic restorations. It presented robust clinical evidence that airborne particle abrasion with alumina particles at a moderate pressure of 0.1-0.25 MPa and the application of primers or luting composites containing phosphate monomers created lasting bonding to zirconia ceramic under humid, stressful oral conditions [4].

Ceramic bonding surfaces conditioned by airborne-particle abrasion are compromised by clinical try-in, which inevitably involves contamination with saliva, blood, and silicone disclosing agents [5]. The supramolecular protein aggregates, specifically proline-rich proteins, have the potential to bind to substrates through a variety of forces mediated by physicochemical interactions between solid substrates and saliva [6,7]. Zirconia and glass ceramics in the oral cavity possess various polarities and physical features, which influence the adsorption of salivary proteins [8]. Saliva is adsorbed onto the surface of zirconia via noncovalent bonds [9]. A study identified the pellicle components on the zirconia surface after salivary

contamination [10]. This organic salivary coating comprises nitrogen, oxygen, and carbon, and the organic constituents cannot be eliminated by water rinsing alone [9]. Consequently, residual organic and inorganic contamination significantly decreases the bond strength [8,9,11,12].

Wettability is a consequence of molecular interactions that transpire between the adhesive and substrate, in addition to the cohesive forces exhibited by the adhesive, particularly its surface tension characteristics. The phenomenon of wetting is typically assessed through the measurement of the contact angle (θ), which is defined as the internal angle formed between the liquid interface and the substrate surface. In general, diminutive contact angles are observed when a liquid with low surface tension is deposited onto a substrate characterized by high surface energy. Contact angles that measure less than 90 degrees signify a favorable tendency for wetting to occur on the surface. The presence of surface roughness enhances the wettability of the substrate by various liquids [13].

To create long-lasting bonding in spite of former contamination, ample research has been performed on cleansing and eliminating residual contaminants by water spray [14,15], zirconia primer [6], Ivoclean [14], plasma [12], 70%-96% isopropanol alcohol [12], or additional airborne particle abrasion (Al_2O_3) [11,16]. The airborne-particle abrasion technique emerged as the most efficacious among the various cleaning techniques, yielding a bond strength analogous to that observed in uncontaminated control samples following 150 days of immersion in water and exposure to 37,500 thermal cycles [9,17]. Hence, conditioning of the bonding surface of zirconia dental restorations through airborne-particle abrasion was recommended in 2009 to be performed right before cementation, rather than before the clinical try-in, establishing this procedure as the gold standard [17].

Zirconia restorations with complicated surface geometry might complicate the elimination of contaminants with airborne-particle abrasion [17]. Sandblasting can also cause defects, cracks, and surface damage.

Thus, the mechanical properties of zirconia can be compromised [18]. Furthermore, it might not be possible to have access to abrasion devices in dental offices, which makes this phase hard to carry out following the clinical try-in. Therefore, applying a cleaning solution or paste on the bonding surface of a contaminated restoration is an easier cleaning method to achieve durable bonding. Different studies have evaluated the bond strength of zirconia by assessing the effect of various chemical solutions on saliva-contaminated zirconia compared to airborne-particle abrasion [10,13,17].

Clinicians utilize 70% ethanol, a well-known disinfectant [19]. ZirClean (Bisco Inc/Schaumburg/IL/USA) is a non-abrasive cleaning gel used to clean the zirconia bonding surfaces and other prosthetic restorations following intraoral try-in. An alkaline solution like ZirClean, containing potassium hydroxide (KOH, pH of 13), is required for successful elimination of ionic bonds. ZirClean yields reliable results for adhesive cementation by eliminating the phosphate contamination of zirconia ceramic and metal restoration surfaces during try-in.

A substitute technique uses nonthermal atmospheric plasma (NTAP) for surface decontamination [20]. NTAP has minimal toxic effects [21], leaves no residue, has no effect on surface morphology [22], and may be useful for maintaining the mineral content [23]. Two mechanisms of action are suggested for NTAP, including the production of reactive species like nitrogen, oxygen, or nitrogen oxide radicals which promote higher surface energy [24] and the oxidation-induced etching impact [25].

Therefore, the information available on the effect of NTAP on bonding procedures shows its limited application in clinical dentistry. Yet, *in vitro* research has documented the biological and chemical impact of NTAP [26] and its various applications such as decontamination through deactivation of oral pathogens, reduction of microbial adhesion, and formation of biofilm on surfaces [11] as a surface agent modifier, enhancing the surface wettability of dentin [27] or composite resin [28]. Research has explored the impact of

NTAP for surface treatment and elimination of salivary contamination.

However, the impact of NTAP and ZirClean on blood contamination has not been addressed. Therefore, this study was conducted to examine and compare the impact of various cleaning techniques on microshear bond strength (μ SBS) of contaminated yttria-stabilized tetragonal zirconia polycrystal ceramic to self-adhesive resin cement, and surface wettability. The null hypotheses were that different cleaning methods 1) would not affect the μ SBS of contaminated yttria-stabilized tetragonal zirconia polycrystal ceramic to self-adhesive resin cement, and 2) would not affect the surface wettability (contact angle).

MATERIALS AND METHODS

Specimen preparation:

The sample size for each experimental group was determined through power analysis to ensure the study had sufficient statistical power to detect meaningful differences between the tested methods. Based on a significance level (α) of 0.05 and a statistical power ($1-\beta$) of 0.80, and assuming a standard deviation (σ) of 3 and a minimum detectable difference (d) of 3MPa, the sample size (n) was calculated using the following formula:

$$n = \frac{\left(Z_{1-\frac{\alpha}{2}} + Z_{1-\beta} \right)^2 (\sigma_1^2 + \sigma_2^2)}{d^2}$$

Applying these parameters, it was determined that 16 specimens per group were required for microtensile bond strength test. This sample size was deemed adequate to reliably evaluate the bonding performance of the zirconia ceramics under the specified experimental conditions. Additionally, five specimens per group were designated for contact angle measurements, resulting in a total sample size of 126 specimens (Fig 1).

The square-shaped zirconia specimens (inCoris ZI; Sirona Dental Systems, Bensheim, Germany) with 10mm×10mm dimensions and 3mm thickness were used in this *in vitro* study.

3Y-TZP (n=126)					
Abraded with 50µm Al ₂ O ₃ at a pressure of 0.2MPa					
Uncontaminated (n=21)	The discs (n=105) immersed in saliva for 60s → rinsed with distilled water for 15s → dried with air for 15s → immersed in blood for 60s → rinsed with distilled water for 15s → dried with air for 15s.				
UN No additional cleaning (n=21)	CW Rinsed with distilled water (n=21)	CE 70% Isopropanol ultrasonic bath for 3 minutes (n=21)	SHC 1% NaOCl apply and rinse with water (n=21)	CZ Zirclean According to the manufacturer's instructions (n=21)	NTAP Air: 5 minutes plasma treatment (n=21)
Contact angle test by sessile drop method (n=5/group)					
Specimens (n=16/group) were bonded with Thracem and TBS was measured after 7 days					

Fig 1. Study design for surface contact angle and bond strength testing.

First, the specimens were polished with 600-grit carbide abrasive papers under water irrigation, after which they were cleaned in a 70% isopropanol alcohol ultrasonic bath for 3 minutes. Then, they were horizontally and vertically airborne-particle abraded with 50µm Al₂O₃ particles at a pressure of 2 bar for 15 seconds at a distance of 10mm from the bonding surface to ensure that the entire surface was abraded. Next, the samples were cleaned in a 70% isopropanol ultrasonic bath for 3 minutes.

The specimens were then randomly grouped based on the contamination and cleaning techniques. Fresh saliva of a healthy donor who abstained from eating and drinking for 1.5 hours before collection was used for contamination, and blood was taken from the same person's fingertip (confirmed by the Institutional Ethics Committee, CAAE: IR.MUI.RESEARCH.REC.1399.728). The specimens were contaminated through immersion in fresh saliva for 60 seconds at ambient temperature, subsequently rinsed

with distilled water for 15 seconds, and then dried utilizing oil-free air stream. Following this procedure, the specimens were immersed in blood for 60 seconds at room temperature, thereafter rinsed with distilled water for 15 seconds, and dried with oil-free air stream. The working area was meticulously cleaned with tap water to guarantee elimination of residual contaminants. Consequently, the specimens were categorized into six experimental groups (n=16/group).as illustrated in Figure 1:

-UN: Uncontaminated, no additional cleaning (control group).

-CW: Contaminated, rinsed with distilled water for 15 seconds, and dried with an oil-free air stream.

-CE: Contaminated, cleaned in 70% ethanol (Pars Alcohol Co/Isfahan/Iran) in an ultrasonic bath (Behin Tamin Ahoora Co/Isfahan/Iran) for 3 minutes.

-SHC: Contaminated, cleaned with 1% sodium hypochlorite solution (Raga/Tehran/Iran) for 60 seconds, rinsed

with distilled water for 15 seconds, and dried with oil-free air for 10 seconds.

-CZ: Contaminated, cleaned with ZirClean based on the manufacturer's protocol.

-NTAP: Contaminated, rinsed with distilled water for 15 seconds, dried with oil-free air, and then placed in the plasma chamber for the final cleaning process with air atmospheric plasma for 5 minutes (Enhancedtech-16I; Kavoshyaran, Iran).

Then, resin cylinders were produced uniformly by injecting TheraCem self-adhesive resin cement (Bisco

Inc/Schaumburg/IL/USA) into a prefabricated plastic Tygon tube (Norton Performance Plastic; Cleveland, OH, USA) with 0.8mm diameter and 2mm height, and bonded to the conditioned zirconia surfaces. It was then light-polymerized by a light-emitting diode curing unit (VALO LED light curing unit; Ultradent Products Inc., South Jordan, UT, USA) at 1000–1200mW/cm² intensity for 20 seconds. The specimens were kept in deionized water at 37°C for 1 week before the μ SBS test. Table 1 presents the specifications of the materials utilized in this research.

Table 1. Materials used in this study and their chemical compositions and instructions for use

Product	Company	Composition	Instructions
TheraCem (Self-Adhesive Resin Cement)	Bisco Inc., Schaumburg, IL, USA	10-MDP, Portland cement, Ytterbium fluoride, Barium glass, Dimethacrylate resins	Apply the resin cement and light-cure it for 20 seconds.
InCoris ZI Block	Sirona Dental Systems, Bensheim, Germany	Zirconium dioxide + hafnium dioxide + yttrium trioxide >99 wt.%, aluminum trioxide <0.5 wt.%, others oxides <0.5 wt.%	Sinter (1550°C/2 hours). Sandblast with 50 μ m alumina for 15 seconds (air pressure: 0.2MPa; distance from the tip: 10mm) and ultrasonically clean (5 minutes)
Ethanol	Nasr Alcohol, Iran	70% Ethyl alcohol (C ₂ H ₅ OH) + 30% Water (H ₂ O)	Clean in 70% ethanol in an ultrasonic bath for 3 minutes
Sodium hypochlorite solution	Raga, Iran	Potassium hydroxide (KOH, pH: 13)	Clean with 1% sodium hypochlorite solution for 60 seconds, rinse with distilled water for 15 seconds, and dry with oil-free air for 10seconds.
ZirClean (cleaning gel)	Bisco Inc., Schaumburg, IL, USA	potassium hydroxide (KOH, pH: 13)	1. After try-in, thoroughly rinse the restoration with water spray and dry with oil-free air. 2. Cover all bonded surfaces of the restoration with a layer of ZirClean. 3. Allow 20 seconds for the cleaning action of ZirClean to take effect, then thoroughly rinse with water spray and dry with oil-free air. 4. Sandblast the internal surface of restoration (unless the restoration has previously been sandblasted). Rinse with water spray and dry the surface with oil-free air.
Nonthermal atmospheric plasma (NTAP)	Enhancedtech-16I, Kavoshyarn, Iran	Air atmospheric plasma	Place it in the plasma chamber for a final cleaning process with air atmospheric plasma for 5 minutes.

The specimens were fixed in an acrylic resin cylindrical mold and connected to a shear-testing device. Measurements were conducted by a universal testing machine (K21046; Walter & Bai, Lohningen, Switzerland). Shear load was applied to the zirconia-resin cement interface with a crosshead speed of 0.5mm/min until failure occurred. Bond strength was computed by dividing the peak load causing failure by the surface area of the specimen, and the μ SBS means were obtained in megapascals (MPa). One-way ANOVA and Tukey's post-hoc test were used to analyze the bond strength data ($\alpha=0.05$).

Failure mode:

After the μ SBS test was conducted, the failure mode of the specimens was determined by examining the interfacial zone under a stereomicroscope (MBC-10, SF-100; Lomo, St. Petersburg, Russia). Failure modes were grouped into (a) adhesive failure: between the zirconia surface and resin cement, (b) cohesive failure: within the resin cement, and (c) mixed: a combination of adhesive failure at the interface and cohesive failure within resin.

Contact angle by the sessile drop method:

Zirconia plates (n=5) were prepared to calculate the contact angle by a goniometer (CA-500A; Irasol, Iran) using the dynamic sessile drop technique. The sandblasted zirconia plates were placed on the metal platform of the device and leveled. The same process of decontamination was used for this test. The angle created by the deionized water on the specimen surface was computed 5 seconds after drop placement.

Statistical analysis:

Since normal distribution of data in the groups was confirmed by the Kolmogorov-Smirnov test and homogeneity of the variances was confirmed by the Levene's test, data were statistically analyzed by ANOVA and Tukey's post-hoc test ($\alpha=0.05$).

RESULTS

μ SBS and failure mode:

ANOVA showed a significant difference in μ SBS among the study groups ($P<0.001$). The Tukey's test was also used for pairwise comparisons. Table 2 shows the μ SBS results of the test groups.

The SHC and NTAP groups produced the highest μ SBS, which did not significantly differ from the μ SBS in the NC group. WS created the lowest μ SBS, which was not significantly different from the CE and CZ groups. The failure modes are presented in Table 3.

Contact angle:

ANOVA showed a significant difference in contact angle among the study groups ($P<0.001$). The Tukey's test was also performed for pairwise comparisons, and the results are summarized in Table 4.

CE produced the highest contact angle, which was not significantly different from the NC and CZ ($P>0.05$), and SHC showed the lowest contact angle. There was no significant difference in contact angle between the NTAP and CW ($P>0.05$), and the contact angle of NTAP was significantly higher than that of SHC ($P=0.014$).

Figure 2 illustrates the representative contact angle images of each group.

Table 2. Descriptive statistics of the μ SBS (MPa) in the experimental groups (n=16)

Group	Mean	SD	SE	95% CI		Min	Max
				Lower Bound	Upper Bound		
NC	12.44 ^{a,b}	3.95	99	10.34	14.55	5.82	21.64
Water	7.24 ^c	2.69	0.72	5.69	8.79	2.48	11.41
Ethanol	9.65 ^{b,c}	3.55	0.89	7.76	11.55	2.20	14.07
SHC	13.56 ^a	3.31	0.83	11.80	15.32	8.59	22.89
Zir	9.64 ^{b,c}	2.91	0.73	8.09	11.19	2.49	13.84
NTAP	10.44 ^{a,b}	2.53	0.63	9.09	11.79	3.57	14.24

NC: Not contaminated; Water: distilled water; Ethanol: 70% ethanol; SHC: 1% sodium hypochlorite solution; Zir: Zirclean; NTAP: nonthermal atmospheric plasma; CI: Confidence Interval for the Mean; SD: Standard Deviation, SE: Standard Error; Min: Minimum; Max: Maximum; Data with different lowercase letters have significant differences ($P<0.05$, Tukey's test).

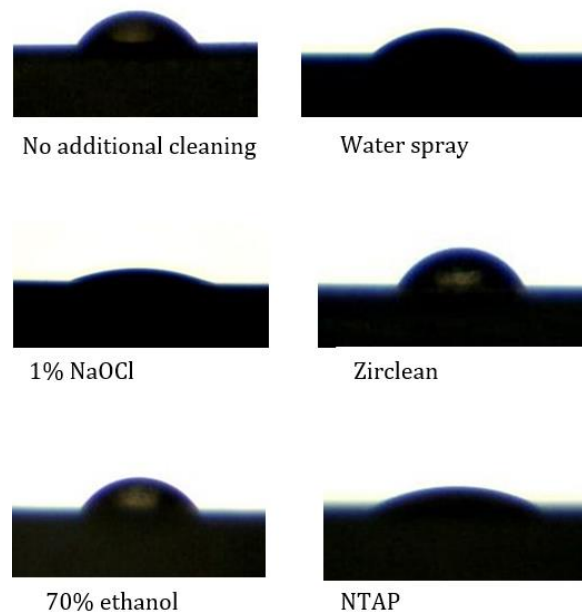
Table 3. Frequency percentage of failure modes for each decontamination protocol

Group	Adhesive	Mixed	Cohesive	Total
Not contaminated	12	4	0	16
Distilled water	14	2	0	16
70% Ethanol	13	3	0	16
1% Sodium hypochlorite solution	9	7	0	16
ZirClean	12	4	0	16
Nonthermal atmospheric plasma	10	6	0	16

Table 4. Descriptive statistics of the contact angle (degrees) in the experimental groups (n=5)

Group	Mean	SD	SE	95% CI		Minimum	Maximum
				Lower Bound	Upper Bound		
NC	57.74 ^c	6.67	2.98	49.45	66.03	48.09	66.57
Water	37.54 ^b	6.37	2.85	29.62	45.45	30.33	44.01
Ethanol	62.32 ^c	1.99	0.89	59.85	64.79	60.51	65.34
SHC	19.54 ^a	4.56	2.04	13.87	25.21	12.92	24.20
Zir	61.00 ^c	8.75	3.91	50.13	71.87	46.09	67.47
NTAP	32.51 ^b	1.14	0.51	31.10	33.92	31.03	34.21

NC: Not contaminated; Water: Distilled water; Ethanol: 70% Ethanol; SHC: 1% Sodium hypochlorite solution; Zir: ZirClean; NTAP: Nonthermal atmospheric plasma; CI: Confidence Interval for the Mean; SD: Standard Deviation, SE: Standard Error; Data with different lowercase letters have significant differences ($P < 0.05$, Tukey's test).

**Fig 2.** Representative images of the contact angle after each decontamination protocol.

DISCUSSION

Bonding to zirconia is challenging. Since zirconia is a polycrystalline ceramic with no glass phase, no optimal cementation protocol has been proposed for it; thus, hydrofluoric

acid etching or silanization is not suggested [29]. The hypothesis of this research was partly rejected. The surface treatment of zirconia is frequently done by airborne particle abrasion [30]. In the present study, all specimens were sandblasted before salivary contamination. The air-borne particle abrasion-induced roughness elevated the salivary adsorption and challenged the potential efficacy of the cleaning agents [31]. The chemical cleaning agents that can be easily used and are readily available in dental offices were examined in this study, and no additional sandblasting was applied following contamination. Although air-borne particle abrasion after salivary contamination produced the highest cleaning efficacy [31], it needs a distinct clinical procedure, and since multiple intraoral try-ins are required, this process is less applicable in daily practice. Furthermore, loose alumina particles may remain on bonding surfaces, and negatively influence the bonding durability in the long run [16]. As for the impact of air-borne particle abrasion on zirconia bond strength, conflicting reports have been presented [16]. Particle

abrasion can cause cracks on the zirconia surface and can degrade the bond strength [32]. Therefore, the use of air-borne particle abrasion for the second time to clean contaminated surfaces can bring about unanticipated results; thus, it was not employed in this study.

Wetting is typically assessed through the measurement of the contact angle (θ), defined as the internal angle formed between the liquid interface and the substrate. In general, diminutive contact angles are attained when a liquid exhibiting low surface tension is deposited on a substrate characterized by high surface energy. Contact angles measuring less than 90 degrees signify a favorable interaction of surface wetting. Optimal wetting is realized when the liquid uniformly disperses across the surface, with θ approximating 0 degrees. The presence of surface roughness enhances the wettability of the substrate by liquids [12]. The self-adhesive resin cement (Theracem) employed in this study contains 10-MDP, a hydrophobic aromatic dimethacrylate and hydrophobic aliphatic dimethacrylate functional group. The 10-MDP-based resin cements are often proposed for zirconia cementation. The phosphate ester group of 10-MDP is bonded to zirconium oxide [3]. Yet, this bonding is not the only mechanism for the resin-containing 10-MDP [33]. The application of airborne particle abrasion along with 10-MDP-containing resin creates long-lasting bond strength values *in vitro* [33]. Therefore, a self-adhesive cement containing 10-MDP was applied to lute the zirconia blocks abraded with airborne particles.

In this study, the μ SBS values of contaminated zirconia cleaned with water were lower compared to the non-contaminated group, which confirms prior findings [34]; thus, this technique cannot significantly decrease the impact of blood and saliva contamination. To obtain higher μ SBS values, complete removal and a better disinfection method are required. The use of 70% ethanol was another disinfecting procedure used in this study. This solution can induce cell lysis and denaturation of bacterial proteins [19], and applying 70% ethanol on the zirconia surface before cementation can be a substitute method for

surface decontamination. However, in this study, no difference was found between CE and CW in μ SBS values. This might be because 70% ethanol could not eliminate all the organic materials deposited on the zirconia surface completely.

To discover the effectiveness of various cleaning procedures and to better comprehend the bond strength findings, previous studies examined the zirconia surface for adherent organic components using x-ray photoelectron spectroscopy. Carbon (C), oxygen (O), nitrogen (N), and zirconium (Zr) elemental ratios were determined. They reported increasing C:Zr, O:Zr, C:O, N:Zr, and N:O elemental ratios in the contaminated samples that could not be eliminated with distilled water rinsing for 15 seconds or by 99% isopropanol ultrasonic bath for 3 minutes [8,34]. N was also detected in the samples contaminated with saliva and blood [34], but it was not found in samples merely contaminated with saliva [8].

The decreased bond strength and elevated frequency of adhesive failures in the CW and CE groups can be attributed to the presence of organic residue, which is chiefly composed of oxygen, nitrogen, and carbon, spotted by x-ray photoelectron spectroscopy on the zirconia surface. This residue inhibits the phosphate monomer-zirconia ceramic chemical bonding [34].

According to a recent report, for a 60-second application time, higher sodium hypochlorite concentrations elevated the dentin-composite resin bond strength to a peak at 10% concentration [35]. Although 5.25% sodium hypochlorite is a tissue solvent that is frequently utilized, 1% sodium hypochlorite solution can efficiently induce tissue dissolution [36]. The experimental zirconia cleaning solutions with a rather low concentration (1.0wt%) were produced and examined owing to their potential application in intraoral repair procedures using composite resin. Despite the fact that zirconia is relatively hydrophobic and has a low surface free energy [37], sodium hypochlorite generated the lowest contact angle and increased micro-retention, confirming the results of a former research [17]. The residual sodium hypochlorite

is also known to interfere with resin polymerization because of oxygen generation [38]. However, 1% sodium hypochlorite was the most efficient solution among the experimental cleaning solutions to eliminate salivary contaminants from the zirconia surface. The x-ray photoelectron spectroscopy analysis in another study also indicated a lower O/Zr ratio for the sodium hypochlorite-cleaned zirconia surface than H₂O₂-cleaned area. Scanning electron microscopic analysis of the debonded surface showed little interfacial gaps [17]. These results might imply that sodium hypochlorite effectively cleansed the surface, and then water-spraying mostly eliminated the residual sodium hypochlorite from the zirconia surface [15,17].

ZirClean, an alkaline solution containing potassium hydroxide (KOH, pH of 13), enhances the bonding of MDP monomers to the TheraCem cement by eliminating the phosphate contamination of zirconia. No significant difference was found between the μ SBS values of CE and those of CW and CE groups, indicating that ZirClean cannot completely eliminate the contaminants.

As for NTAP, similar μ SBS values and decreased contact angle were found compared to the control group, confirming the results of previous research [11]. Application of plasma on the surface may eliminate the organic residues, thereby enhancing chemical restructuring of the surface and decreasing bacterial survival [20]. Other features of NTAP are reducing carbon and elevating oxygen [22], elevating the oxygenic polar groups, and enhancing a more hydrophilic surface. Since zirconia is a hydrophobic substance with no surface density of hydroxyl groups, it does not create a strong bonding to resin. The application of plasma enhances the surface wettability, thereby creating better bonding to resin [27], which confirms the results of the present study. Plasma treatment does not influence the surface morphology despite these modifications; NTAP only affects the wettability [22].

The lowest and the highest rates of adhesive failure between the zirconia and the luting agent were reported in the SHC and CW

groups, respectively, suggesting that sodium hypochlorite can effectively clean zirconia.

The contact angle of all groups was less than 90 degrees. There was no association between the contact angle and bond strength of the groups except in the SHC group. Sodium hypochlorite created the highest μ SBS and lowest contact angle. No statistically significant difference was found between the contact angle of the CZ group and that of UN and CE groups, indicating that ZirClean and ethanol do not affect the zirconia surface hydrophilicity.

The present research only reported the results of one-week bond strength. Further studies are needed to find out whether the decontamination protocols can affect the long-term μ SBS values. Further research is also required to examine the impact of other resin cements and various gases for plasma following long-term water storage with thermocycling.

CONCLUSION

Within the restrictions of the present study, the following conclusions were drawn:

- Sodium hypochlorite could clean the zirconia surface, presenting μ SBS values higher than the contaminated group.
- The 70% ethanol, ZirClean, and NTAP decontaminated the saliva- and blood-contaminated zirconia surfaces.
- Sodium hypochlorite elevated the wettability of the zirconia ceramic surface; whereas, 70% ethanol, and ZirClean did not affect the contact angle compared to the non-contaminated group.

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CONFLICT OF INTEREST STATEMENT

None declared.

GENERATIVE AI IN SCIENTIFIC WRITING

None declared.

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