Effect of Cleaning Methods on the Micro-shear Bond Strength of Zirconia Contaminated with Disclosing Silicones

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Abstract

Zirconia is one of the most commonly used materials for indirect dental restorations. Contaminated with disclosing silicone leads to a decrease in bond strength between zirconia and resin cement. The Proposal of this study was to investigate the effect of different cleaning methods on microshear bond strength (µSBS) between disclosing silicone-contaminated zirconia and resin cement. The 168 cylindrical plates of Cercon®ht zirconia were fabricated and randomly divided into 4 groups according to contamination agents (Non-contaminated (CTRL), FIT CHECKER™ ADVANCED (FC), TOKUYAMA FIT TESTER (TFT), and coltène® PSI (PSI)). The specimens in each group were assigned to 6 subgroups with different cleansing procedures: water rinsing (W), air abrasion (AA), Ivoclean® (IC), 70% ethanol (EN), 37% phosphoric acid (PA), and 4.5% hydrofluoric acid (HF). Two specimens from each group underwent surface morphology examination and contact angle measurement. The remaining specimens were applied with CLEARFIL™ CERAMIC PRIMER PLUS, cemented with Multilink® N resin cement and stored in 37°C distilled water for 24h, then subjected to µSBS test using a universal testing machine. The data were analyzed by Two-way ANOVA and Tukey's test ($p<0.05$). Failure modes were categorized using a light stereomicroscope. Disclosing silicone contamination resulted in a significant reduction of µSBS regardless of the type of disclosing silicone. After decontamination, the highest µSBS was found in AA groups. Mixed failure was predominantly found in all groups. The µSBS between resin cement and zirconia was significantly affected by disclosing silicone contamination. Air abrasion is the most effective surface cleaning method for regaining µSBS.

Keywords: *zirconia, disclosing silicone, cleaning methods, resin cement*

1. Introduction

Zirconium dioxide (ZrO_2), or zirconia, is a type of all-ceramic restorative material classified as polycrystalline ceramic, have become increasingly popular for prosthetic restorations in dentistry to restore the function of compromised teeth due to its high mechanical properties, esthetic appearance, and biocompatibility (Gracis et al., 2016). Zirconia can be used in both anterior and posterior teeth, replacing full metal and porcelain fused-to-metal restorations (Nistor et al., 2019).

Due to its high strength and fracture resistance, the soft machining technique is typically used to fabricate zirconia. They utilize oversized pre-sintered zirconia, with compensation for shrinkage already calculated before final sintering using Computer-Aided Design (CAD), to prevent stress-induced phase transformation from tetragonal to monoclinic, which could exacerbate surface microcracking and low-temperature degradation (Nistor et al., 2019).

Even though the software has become more precise nowadays, evaluating the adaptation of the restoration prior to bonding remains a critical step, as any marginal discrepancies on the proximal surfaces, subgingival areas, and internal gaps cannot be detected through visual or tactile inspection alone (Habib et al., 2020). This can be achieved by using disclosing silicone, which is highly preferred due to its ease of use and quick setting time. However, it may lead to contamination on the intaglio surface of the restoration (Yang et al., 2007; Wille et al., 2015).

Manufacturers have claimed that disclosing silicone fully polymerizes after mixing, leaving no residue on the restoration surface. Nevertheless, studies have found that when restorations are contaminated with disclosing silicone, residues consistently remain on the surface (Yang et al., 2007; Quaas, Yang, & Kern, 2007; Wille et al., 2015). Furthermore, zirconia specimens, which are often pre-treated in dental laboratories, tend to exhibit surface roughness that allows more silicone residues to adhere. The remaining residue may act as a barrier, leading to a significant reduction in bond strength.

Therefore, the cleanliness of the surface after evaluating the adaptation is essential for achieving reliable bond strength between zirconia restoration and resin cement. This study investigated the effect of different cleaning methods on µSBS between zirconia-contaminated disclosing silicones and resin cements.

The null hypothesis was that there was no difference in µSBS between zirconia contaminated disclosing silicones and resin cements using different disclosing silicones and different cleaning methods.

2. Objectives

1) To investigate the effect of contamination from different types of disclosing silicone on the microshear bond strength between resin cement and zirconia.

2) To investigate the effect of different cleaning methods on the microshear bond strength between disclosing silicone contaminated zirconia and a resin cement.

3. Materials and Methods

One hundred and sixty-eight cylindrical plates of Cercon® ht (Dentsply Sirona, USA) zirconia (10.0 mm diameter x 4.0 mm thickness) were used as substrates. Each plate was embedded in the metal mold with epoxy resin. The bonding side was ground and polished using 800, 1000, 1200, 1500 and 2000 grit silicon carbide paper with grinding machine (MoPao 160E, MEGA Advance, Shandong, China) 1 min per each grit and ultrasonically cleaned in distilled water for 10 min, then sandblasted with 50 µm alumina particles at 2.5 bar for 15 s at 10 mm distance and ultrasonically cleaned again for 10 min and air-dried, specimens were randomly divided into 4 groups, according to contamination agents as follows:

Group 1 (CTRL): The specimens were not contaminated (control group)

Group 2 (FC): The specimens were contaminated by FIT CHECKER™ ADVANCED (GC Corporation, Tokyo, Japan)

Group 3 (TFT): The specimens were contaminated by TOKUYAMA FIT TESTER (Tokuyama Dental Corp, Tokyo, Japan)

Group 4 (PSI): The specimens were contaminated by coltène® PSI (Coltene/Whaledent AG, Switzerland)

The specimens in FC, TFT, and PSI groups were contaminated with disclosing silicone under 1 kg pressure for 4 min, then rinsed with water spray for 1 min and gently dried. Then the specimens in each group were assigned to 6 subgroups according to cleaning procedures as follows:

Group 1 (W): No additional cleaning (only water rinsing after contamination agent removal)

Group 2 (AA): Air abrasion with 50 µm alumina particles at 2.5 bar for 15 s at 10 mm distance and ultrasonically cleaned for 10 min and air-dried.

Group 3 (IC): Ivoclean® (Ivoclar Vivadent, Schaan, Liechtenstein)

Group 4 (EN): 70% ethanol

Group 5 (PA): 37% phosphoric acid (Meta Etchant, Meta Biomed®, Chungcheongbuk-do, Korea)

Group 6 (HF): 4.5% Hydrofluoric acid (IPS® Ceramic Etching Gel, Ivoclar Vivadent, Schaan, Liechtenstein)

In IC, EN, PA and HF groups, the cleaning agents were applied and agitated with a microbrush for 20 s, rinsed with water spray for 10 s and gently dried with oil-free air for 10 s.

After the surfaces were treated using the above methods, two specimens from each group were randomly selected for surface morphology examination by using a scanning electron microscope (SEM) and water contact angle measurement by using a goniometer (KINO SL250, KINO Scientific Instrument Inc., USA) to evaluate hydrophobicity. The other 5 specimens were primed with CLEARFIL™ CERAMIC

PRIMER PLUS (Kuraray Noritake Dental Inc., Japan), an MDP-containing surface priming agent, for 1 minute and dried with oil-free air. Four Tygon tubes (Saint Gobain Performance Plastics, USA) with 0.8 mm internal diameter and 0.5 mm height were placed over each specimen with a clamping device. Freshly mixed Multilink® N dual-cured resin cement (Ivoclar Vivadent, Schaan, Liechtenstein) was injected into the iris of each tube and light cured with Bluephase N® LED Curing Light (Ivoclar Vivadent, Liechtenstein) in soft-start mode for 20 s. The bonded specimens were stored in distilled water at 37 °C for 24 h, and then the µSBS was measured using a universal testing machine (UTM) (Instron® 5566 universal testing machine, Instron Engineering Corporation, Massachusetts, USA). A micro-shear force was applied with a 100 N load cell, crosshead speed 1 mm/min.

Table 1 Materials used in this study

Product names and manufacturers	Composition	Batch number
Multilink® N (Ivoclar Vivadent, Liechtenstein)	Base: Dimethacrylate, HEMA, organic filler, tertiary amine Catalyst: Dimethacrylate, HEMA, filler, dibenzoyl peroxide	Z0347C
CLEARFIL™ CERAMIC PRIMER PLUS (Kuraray Noritake Dental Inc., Japan)	Ethanol > 80%, 3-MPS < 5%, 10-MDP	BG0088
Ivoclean® (Ivoclar Vivadent, Schaan, Liechtenstein)	Zirconium oxide 10-15%, Water 65-80%, Polyethylene glycol 8-10%, Sodium hydroxide ≤ 1%, Pigments and additives 4-5%	Z03WRY
FIT CHECKER™ ADVANCED (GC Corporation, Tokyo, Japan)	Vinyl polyether addition silicone disclosing agent Base: Silicon dioxide 25-50%, Methyl hydrogen dimethylpolysiloxane 10-25% Catalyst: Titanium dioxide 0.5-1%	2203141
TOKUYAMA Fit Tester (Tokuyama Dental Corp, Tokyo, Japan)	Silica containing addition silicone disclosing agent	172E83
coltène® PSI (Coltene/Whaledent AG, Switzerland)	Condensation silicone elastomer Base: Polysiloxane, Catalyst: Dioctyltin dicarboxylate 5-<10%, Alkyl silicates 15- <20%	L64485
37% phosphoric acid (Meta Etchant, Meta Biomed®, Chungcheongbuk-do, Korea)	Phosphoric acid <40%, Distilled water <60%, Xanthan gum 1-5%, Blue pigment 1-5%	MET2207251
4.5% Hydrofluoric acid (IPS® Ceramic Etching Gel, Ivoclar Vivadent, Schaan, Liechtenstein)	Hydrofluoric acid 4.5%	Z037BV
70% ethanol	Ethyl alcohol 70%	09730498

HEMA = 2-hydroxyethyl methacrylate
3-MPS = 3-methacryloxypropyl trimethoxysilane
10-MDP = 10-methacryloyloxydecyl dihydrogen phosphate

Data were collected and converted to megapascal (MPa) and analyzed using Two-way ANOVA, followed by Tukey's test at a 95% confidence level ($p<0.05$). Modes of failure were observed with a light stereomicroscope and categorized into 3 groups:

Cohesive failure: the failure occurred within resin cement for more than 80% of the bonded area.

Adhesive failure: the failure occurred at zirconia- resin cement interface, for more than 80% of the bonded area.

Mixed failure: the failure occurred at the zirconia-resin cement interface and within cement or ceramic.

4. Results and Discussion

4.1 Results

The surface morphology of zirconia contaminated with different types of disclosing silicone and cleansing methods, as observed by SEM, did not show any distinct surface characteristics between the groups.

Measurement of the water contact angle revealed an increase in the contact angle in all groups. The PSI group exhibited distinct liquid droplet formation, indicating a hydrophobic surface, which was able to be detected by the naked eye.

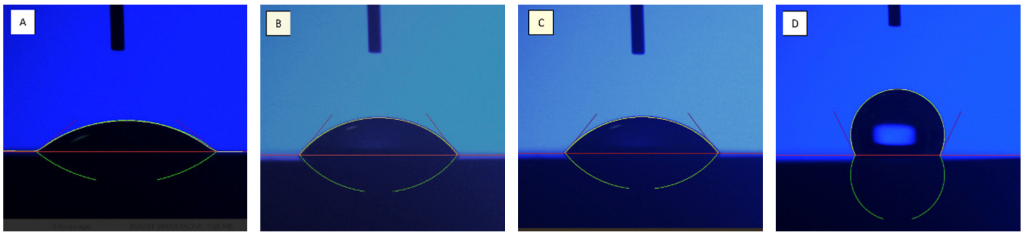

Figure 1 Contact Angle Measurement A. CTRL group; B. FC group; C. TFT group; D. PSI group

After decontamination, there was a decrease in the water contact angle in all groups. The PSI group showed the most noticeable change, as shown in Table 2.

Table 2 Water contact angle measurement (degree)

Group	Decontamination methods	Water Contact Angle (degree)			
		CTRL	FC	TFT	PSI
1	W	38.3	52.5	45.2	119.1
2	AA	36	43.2	40	60.3
3	IC	37.2	47.1	42.2	72.9
4	EN	38	48.3	44.3	75.7
5	PA	41.5	46.6	42.5	68.6
6	HF	39.4	46.3	42.9	76.3

The mean µSBS and standard deviation of each group are shown in Table 3. According to two-way ANOVA, there are no statistically significant differences (p>0.05) in bond strength between each type of disclosing silicone in all groups, while different decontamination methods influenced the bond strength of resin cement to zirconia. The mean µSBS of AA groups were significantly higher (p<0.05) than those of no additional cleaning groups.

Table 3 Mean microshear bond strength and standard deviation of each group (Mean ± SD, MPa)

Group	Decontamination methods	Contaminations			
		CTRL	FC	TFT	PSI
1	W	36.01 ± 6.36 ^{i}	26.76 ± 4.40 ^{cdef}	27.75 ± 2.64 ^{def}	27.63 ± 3.54 ^{def}
2	AA	33.41 ± 4.16 ^{hi}	32.72 ± 3.47 ^{ghi}	32.79 ± 4.77 ^{ghi}	32.98 ± 3.80 ^{hi}
3	IC	33.19 ± 4.87 ^{hi}	30.86 ± 3.85 ^{fgh}	30.45 ± 4.90 ^{efgh}	28.98 ± 4.21 ^{efgh}
4	EN	33.34 ± 4.06 ^{hi}	28.61 ± 4.08 ^{efgh}	28.06 ± 3.47 ^{efg}	28.14 ± 3.43 ^{efg}
5	PA	27.30 ± 4.87 ^{def}	20.60 ± 3.27 ^{a}	23.11 ± 3.64 ^{abcd}	21.86 ± 3.80 ^{ab}
6	HF	30.72 ± 2.91 ^{fgh}	22.45 ± 2.91 ^{abc}	25.89 ± 4.16 ^{bcde}	27.34 ± 4.09 ^{def}

Different superscript letters indicate statistical differences. (p<0.05)

Failure mode analysis showed predominantly mixed failure in every group. Cohesive failure was predominantly found in the CTRL group. Meanwhile, adhesive failure was predominantly found in contaminated groups (Figure 2).

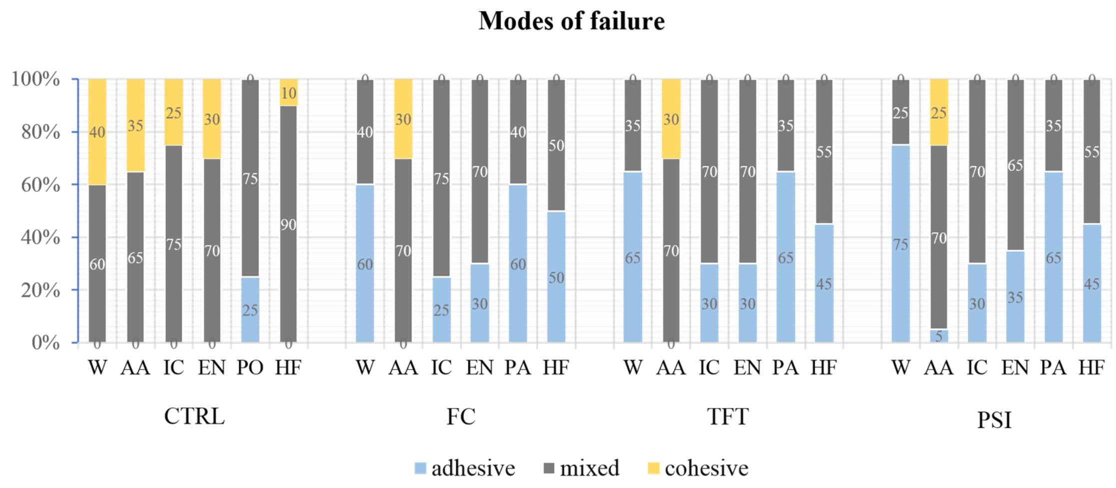

Figure 2 Modes of failure

4.2 Discussion

The results of this study showed that disclosing silicone contamination could alter the hydrophobicity of the surface, resulting in an increased water contact angle. The PSI group exhibited a distinct water droplet shape on the zirconia surface, indicating greater hydrophobicity.

PSI is a condensation silicone. They consist of polydimethylsiloxane (PDMS), which is highly hydrophobic from the inert methyl group (Braden, 1992; Mata et al., 2005). The catalyst actively participates in the condensation reaction between functional groups in monomers. By-products, such as ethanol and water, are released during condensation polymerization reactions (Braden, 1992).

While FC and FTF are addition silicones, they mainly consist of polyvinylsiloxane and polymethylhydrosiloxane, which contain reactive double bonds from vinyl (C=C) and hydroxyl groups (-OH) (Nassar et al., 2018). The catalyst in the addition process primarily initiates the chain reaction by breaking the double bond, after which the reaction propagates indefinitely (Saleh, & Gupta, 2016). When strong carbon-carbon crosslinking occurs, no volatile by-products are formed (McKeen, 2014).

In addition, due to the significant ratio difference between the base and catalyst in the PSI group (10:1.4), compared to FC and FTF (1:1), there is a higher likelihood of inadequate mixing in the PSI group. Improper mixing procedures may result in unreacted monomers remaining in the system. If addition silicone residues remain, they tend to be less hydrophobic compared to condensation silicone residues due to the presence of the hydroxyl group in their composition (Nassar et al., 2018; Singer et al., 2022).

This increase of hydrophobicity confirmed the remains of disclosing silicone residues. After decontamination, there was a decrease in the water contact angle in all groups, with varying degrees. According to a study by Yang et al., (2007), the presence of Si on ceramic after disclosing silicone was peeled off, demonstrated as a residue that created an unstable bond to ceramic restation and could lead to a drastic decrease in bond strength.

In this study, the µSBS results indicated that contamination with disclosing silicone led to a reduction in µSBS, regardless of the type of disclosing silicone use. Water rinsing alone was not sufficient to regain bond strength. The cleaning methods used in this study are commonly used in clinical practice. The µSBS results of the AA group showed that this method was the most effective for decontaminating disclosing silicone. Therefore, the null hypothesis that different disclosing silicones do not affect the µSBS between zirconia and resin cement is accepted. In contrast, the null hypothesis that different cleaning methods do not affect the µSBS is rejected.

Air abrasion with alumina particles is one of the mechanical surface treatment methods for zirconia, which enhances the bond strength between resin cement and zirconia (Łagodzińska, Dejak, & Konieczny, 2023). The result of this study showed that air abrasion can restore the bond strength after beingcontaminated with disclosed silicone. The µSBS was significantly higher than the no-addition cleansing group. This finding could be attributed to the removal of contaminants and exposure of a fresh bonding surface through the mechanical removal of superficial ceramic (Yang et al., 2007), resulting in a high bond strength between resin cement and zirconia. However, repeated sandblasting might induce phase transformation and low-temperature degradation, potentially leading to long-term failure (Irmak et al., 2018).

Commercial cleaning products such as Ivoclean® and 70% ethanol, which is recommended by the manufacturer to be used to clean surfaces after contamination, were not as effective as the air abrasion group. The mean µSBS in both groups was slightly increased but did not fully restore the bond strength to the level of uncontaminated surface and was not significantly different (p>0.05) from no additional cleansing group in all types of disclosing silicone. These findings agreed with Quaas et al., (2007) and Wille et al., (2015) that cleaning methods such as Ivoclean® and isopropanol could reduce the amount but could not completely remove the contaminants.

37% Phosphoric acid is commonly used in clinical practice during surface preparation for dental restorations. It has been proven effective in removing saliva contamination from ceramic surfaces (Lapinska et al., 2019). Therefore, researchers aimed to investigate whether it could also effectively remove disclosing silicone contamination. This study showed that phosphoric acid was not very effective, as the mean µSBS was not significantly different from the group with no additional cleaning. The water contact angle was also slightly lower but not comparable to the control group. Similarly, studies by Yang et al., (2007) and Quaas et al., (2007) reported that etching zirconia contaminated with disclosing silicone using phosphoric acid failed to completely remove silicone residues, as evidenced by the reduction in tensile bond strength.

In the case of zirconia, concerns have been raised that phosphate groups in phosphoric acid may compete with the oxide layer, thereby inhibiting the ability of MDP to form chemical bonds (Angkasith et al., 2016). Phark et al. (2009) reported a significant increase in phosphorus on the zirconia surface after cleansing with phosphoric acid. It was also found in this study that the mean µSBS in the non-contaminated group cleaned with phosphoric acid had significantly decreased. This suggests that phosphoric acid may have a negative effect on the µSBS. This result contrasted with Wattanasirmkit, and Charasseangpaisarn (2019), who said that cleaning with 37% phosphoric acid restored the shear bond strength to the same level as that of the non-contaminated group. Moreover, from this previous study, no phosphorus residues were detected in energy-dispersive X-ray spectroscopy (EDS) (Wattanasirmkit, & Charasseangpaisarn, 2019). Further surface element analysis is required to identify and confirm any unknown residues responsible for the reduction in µSBS.

4.5% hydrofluoric acid is commonly used for surface treatment of ceramics with a silica-based composition (Thompson et al., 2010). Compared to phosphoric acid, hydrofluoric acid has been reported to leave no residue on the restoration surface and does not cause damage to zirconia (Wattanasirmkit, & Charasseangpaisarn, 2019). In this study, when hydrofluoric acid was used to clean zirconia surfaces contaminated with disclosing silicone, the water contact angle was slightly decreased, but the mean µSBS was not significantly different from the group with no additional cleaning ($p > 0.05$); this trend was consistent across all three brands of disclosing silicone. Previous studies have also indicated that hydrofluoric acid does not effectively remove disclosing silicone contamination (Hajjaj, & Alzahrani, 2022).

The modes of failure in this study also supported the bond strength values. In the groups with higher bond strength values, including CTRL and AA groups, mixed and cohesive failure patterns were observed, whereas in the contaminated groups with lower bond strength values, mixed and adhesive failures were predominantly observed. Which may be due to the ineffectiveness of the cleaning methods.

The durability of the resin bond was not evaluated in this study, which might be considered a limitation. Further studies should investigate this aspect by comparing pre- and post-thermocycling bond strength to validate the long-term effectiveness of the treatment procedures. Additionally, this in vitro study was conducted at room temperature. Since temperature may affect the setting properties, the results may differ from those in a clinical setting.

5. Conclusion

1) Disclosing silicone contamination has a negative effect on zirconia- resin bond strength. There was no significant difference in mean μSBS in each type of disclosing silicone.

2) Air abrasion is the most effective cleaning method to remove disclosing silicone and regain bond strength between resin cement and zirconia.

3) Other chemical cleansing agents used in this study were insufficient to eliminate disclosing silicone.

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