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Dr. Marwa Mohamed Wahsh
Department of Fixed Prosthodontics, Faculty of Dentistry, Ain Shams University, Egypt

Dr. Doaa Taha Sayed Taha
Department of Fixed Prosthodontics, Faculty of Dentistry, Ain Shams University, Egypt

Omar Mohamed Mostafa Shaheen
Department of Fixed Prosthodontics, Faculty of Dentistry, Ain Shams University, Egypt

Corresponding Author:
Dr. Marwa Mohamed Wahsh
Department of Fixed Prosthodontics, Faculty of Dentistry, Ain Shams University, Egypt

Effect of surface contamination and cleansing methods on resin bond strength and failure modes of partially stabilized zirconia: An *in vitro* study

Dr. Marwa Mohamed Wahsh, Dr. Doaa Taha Sayed Taha and Omar Mohamed Mostafa Shaheen

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Abstract
Objective: The aim of this study was to evaluate the effect of two surface contaminants and different cleansing agents on the resin bond strength of partially stabilized zirconia.
Method: 80 partially stabilized zirconia samples were randomly divided into two groups (n=40) according to the surface contaminant into Group (S): saliva, Group (D): disclosing agent. Each group was subdivided into three subgroups (n=10) according to the cleansing method into group (V): Ivoclean agent, group (A): air abrasion, group (W): water, and then Groups (SC) and (DC): (surface contamination and no cleaning) (n=20). Bonding was done by applying resin cement to the treated surfaces in all groups. After that, all prepared specimens were thermocycler 5000 cycles, followed by an SBS test and then an assessment of the mode of failure.
Results: Images showed that adhesive failure in the resin interface was more predominant in contaminated groups with no surface cleaning and in groups where water was used as a cleaning agent, while mixed failures were found in groups that used Ivoclean, and Airborne particle abrasion, was more predominant in the latter.
Conclusion: Contaminants adversely affected the shear bond strength of resin cement to the zirconia surface. Airborne particle abrasion appears to be the most effective among the different cleansing methods (SBS 32.25 MPa), followed by Ivoclean (SBS 18.28 MPa), exceeding the minimally acceptable range of SBS for clinical use (15 to 21 MPa). However, SBS was lower in the contaminated non-cleansed or water-cleansed groups, suggesting that cleansing is necessary.

Keywords: Surface contamination, cleansing methods, resin bond strength, partially stabilized zirconia

Introduction

New esthetic materials have been developed because of the expanding demand for esthetic dentistry and the questions surrounding the biocompatibility of dental alloys. These days, metal-based restorations are replaced with ceramic materials, giving a more attractive and esthetic outcome. Since zirconia was first used in dentistry, the applications of metal-free ceramic restorations have grown more successful and reliable.

The advent of CAD/CAM technology has revolutionized zirconia production, dramatically enhancing its ease, efficiency, and accuracy. Zirconia is opaque and less translucent than other ceramics like lithium disilicate; however, recently, this problem has been solved by using zirconia as core material and veneered with more translucent ceramic material and by introducing translucent zirconia to overcome the ceramic veneer fractures (chipping) and fractures of the zirconia substructure.

Because zirconia lacks a glassy matrix, it can be difficult to etch with hydrofluoric acid, making bonding to zirconia challenging. Despite inherent challenges, numerous surface treatments have emerged to optimize zirconia bonding. These include chemical cleaning and bonding-promoting agents, laser etching for increased surface area, alumina coating for chemical bonding, tribochemical silica coating for mechanical and chemical adhesion, silica ceramic coating for enhanced compatibility, and airborne particle abrasion for improved roughness and cleansing [1-4].

The luting cement type, biocompatibility, insoluble nature, and resistance to deterioration influence the long-term success of fixed dental prostheses.

Obtaining a clean surface before applying cement has a crucial effect on bond strength and durability [1]. However, the contaminant-free conditions employed *in vitro* studies often mask the significant impact of clinical realities on resin-ceramic bonding. Saliva, blood, and silicone indicators, during try-in procedures, can significantly decrease the bond strength. Therefore, incorporating clinically relevant contamination into testing protocols is essential for establishing reliable and translatable bonding strategies [2]. Zirconia exhibits a high binding affinity to the phosphate (PO_4^{3-}) group, a prevalent component of salivary and bodily fluids [3].

Disclosing agent is used to check the fitting surface during try in of restorations to identify ill-fitting points in the intaglio surface of the restoration if it's not properly seated on the tooth. Cleaning zirconia intaglio surface after use of disclosing agent is important to avoid any interference with the process of bonding to tooth structure.

While phosphoric acid effectively cleans silica-based ceramics, it falls short when it comes to zirconia [4]. Competitive binding of phosphoric acid's phosphate groups with zirconia's hydroxyl groups displaces adhesive monomers, leading to decreased bond strength. Previous studies recommend airborne particle abrasion with 50 µm aluminum oxide as an effective decontamination method for optimal zirconia-adhesive bonding [5, 6]. Ivoclean, an emerging pre-conditioning agent for zirconia restorations, utilizes a hyper-saturated zirconia particle suspension in an alkaline medium. This formulation creates a targeted chemical gradient at the restoration interface, facilitating the selective adsorption and sequestration of surface contaminants. Subsequent rinsing effectively removes the contaminant particles, preparing the surface for optimal adhesive bonding. However, further investigation is warranted to definitively ascertain the most efficacious and reliable cleansing protocol for high-translucency zirconia.

Statement of the problem

Intraoral try-in of zirconia restorations inevitably leads to surface contamination by saliva, blood, or even disclosing agents. Such contamination significantly compromises the zirconia-resin cement bond strength. Consequently, meticulous surface pre-cleaning prior to cementation becomes paramount for achieving long-lasting and reliable restorations. However, currently, a comprehensive comparison of the efficacy of various zirconia cleaners in removing diverse contaminants is absent, and research investigating the bond strength between contaminated zirconia and dual-cure resin cements remains limited. This lack of scientific evidence necessitates further investigation to establish optimal pre-conditioning protocols and ensure predictable clinical outcomes with high-translucency zirconia restorations.

Aim of the study

The aim of this *in vitro* study was to evaluate the effect of three cleansing methods: Airborne particle abrasion, ivoclean, water.

On the shear bond strength (SBS) between dual-cure self-adhesive resin cement and high translucent zirconia surface contaminated by fresh saliva, disclosing agent.

The null hypothesis was that the SBS of resin cement to contaminated high translucent zirconia surface would not be affected neither by different contamination nor cleansing methods.

Materials and Methods

Materials: In this *in vitro* study, specimens were made of high translucent zirconia, subjected to two contaminants, followed by three different ways of cleansing. The effectiveness of the cleansing methods on the SBS with zirconia was evaluated. The materials used in this study are presented in Table 1.

Table 1: Materials used in the study.

	Material	Brand	Lot number
1.	Zirconia	CERAMILL ZOLID HT+ Amann Girrbach AG, Koblach, Austria.	1906000-31
2.	Dual cure self-adhesive resin cement	Breeze™ Self-Adhesive Resin Cement Pentron clinical technologies, LLC, Walingford, CT, USA	8454731
3.	Fresh saliva	Author's own saliva	
4.	Disclosing agent	Bioclear dual color disclosing agent	715091
5.	Universal cleaning agent	Ivoclean Ivoclar, Vivadent, Lichtenstein	Z0304j

Zirconia

High translucent zirconia is supplied in blanks for CAD/CAM milling available in white shade (Fig 1). It is indicated for crowns and bridges either monolithic or as a core to be veneered by porcelain and crown over implant either screw retained, or cement retained.

Fig 1: Blank of high translucent zirconia

Chemical composition [1]

Table 2: Chemical composition of high translucent zirconia by weight percent

ZrO2 + HfO2 + Y2O3	≥ 99.0%
Y2O3	6, 7 - 7, 2%
HfO2	≤ 5%
Al2O3	≤ 0.5%
Other oxides	≤ 1%

Mechanical properties

Table 3: Mechanical properties of high translucent zirconia

Flexural strength	1100 +/- 150 MPa
Modulus of Elasticity	≥ 200
Vickers Hardness	1300 +/- 200
CTE 25 - 500 °C	10,4 +/- 0,5
Chemical Solubility	< 100

Self-adhesive resin cement

Dual cure self-adhesive universal resin cement [2] (Fig 2).

Fig 2: Dual cure self-adhesive resin cement.

Chemical composition

Table 4: Chemical composition of Resin Cement

Base paste	Catalyst paste
7,7,9(or 7,9,9)-trimethyl-4,13-dioxo-3,14- dioxa-5,12-diazahexadecane-1,16-diyl bismethacrylate	2-[(2-methyl-1-oxoallyl) oxy]ethyl 1,3-dihydro1,3-dioxoisobenzofuran-5-carboxylate
2,2'-ethylenedioxydiethyl dimethacrylate	2-hydroxyethyl methacrylate
Phosphorus pentoxide	Dibenzoyl peroxide

Mechanical properties

Table 5: Mechanical properties of Resin Cement

Flexural strength	48 / 75 MPa
Compressive strength	188 / 236 MPa
Modulus of elasticity	4.9 / 8.4 GPa
Surface hardness	202 / 280 MPa
Radiopacity	2.43 mm Al
Film thickness	18 / - µm
Water sorption	39 / 25 µg/mm3
Solubility	15 -3 µg/mm3

Disclosing agent

Dual color disclosing agent [3]. It is indicated for restorative prosthetic material to check if there is an ill-fitting area (Fig 3).

Fig 3: Dual color disclosing agent

Composition

Table 6: Chemical composition of disclosing solution

Methylhydrogen dimethylpolysiloxane	10-20%
Undisclosed components	80-90%

Universal cleaning agent

The universal cleaning agent [4] is indicated for cleaning the bonding surface of prosthetic materials after try-in procedure (Figure 4).

Fig 4: Universal cleaning paste

Composition

Table 7: Chemical composition of Ivoclean

Zirconium oxide	10-15 wt %
Water	65-80 wt %
Polyethelene glycol	8-10 wt %
Sodium hydroxide	≤1 wt %
Pigments, additives	11-5 wt %

[1] Ceramill zolid ht+, Amann Girrbach AG, Koblach, Austria.
[2] Breeze™ Self-Adhesive Resin Cement Pentron clinical technologies, LLC, Walingford, CT, USA
[3] Bioclear, Tacoma, Washington
[4] Ivoclar, Vivadent, Lichtenstein

Methods

In this *in vitro* study, a total of 80 zirconia square samples were constructed, then tested for shear bond strength of resin cement after being contaminated and cleansed with different cleaning methods. The following diagram is a schematic presentation showing study methodology. (Fig 5).

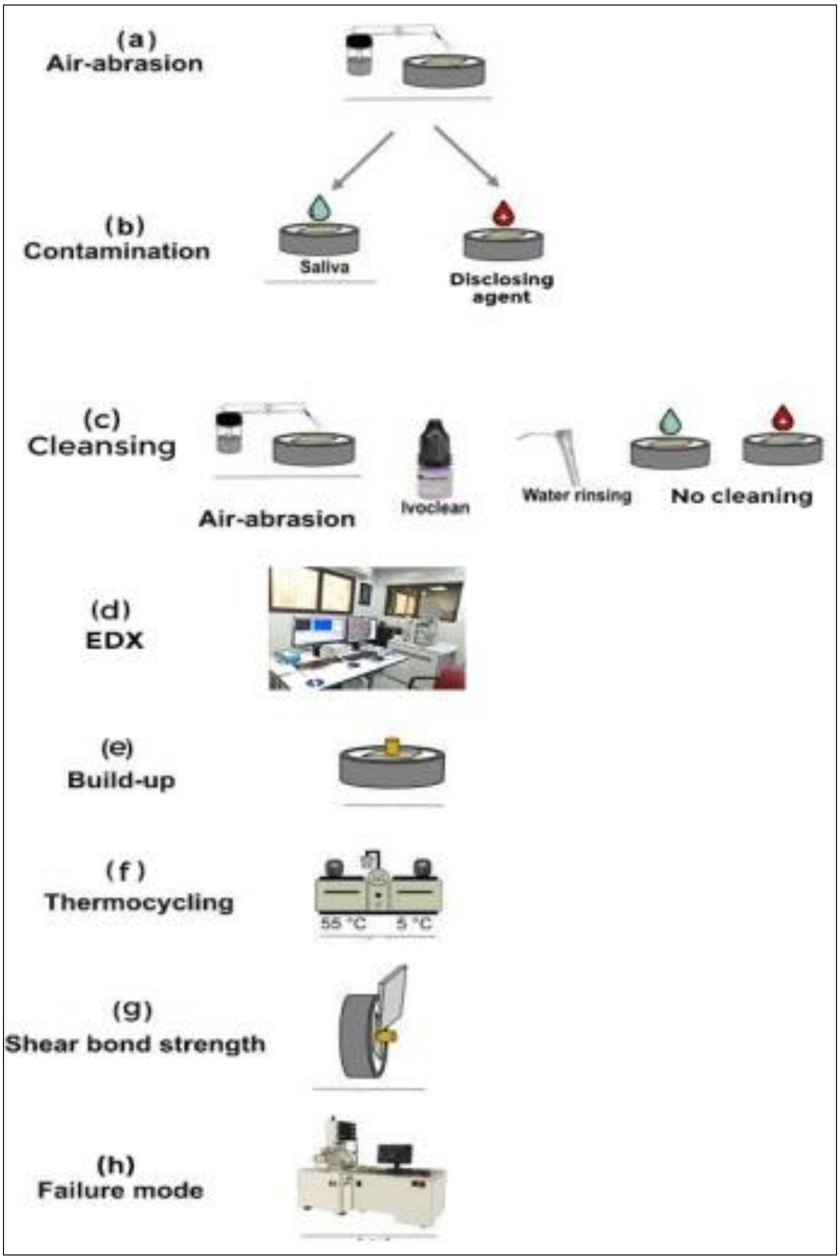

Fig 5: Illustration of the study methodology.

Sample grouping

A power analysis aimed to guarantee sufficient statistical power for testing the null hypothesis of no difference in bond strength across groups. By adopting an alpha (α) level of 0.05 (5%), a beta (β) level of 0.1 (i.e., power = 90%), and an effect size (f) of (0.504) calculated based on the results of a previous study [31]; the predicted sample size (n) was a total of (80) samples. Sample size calculation was performed using G*Power version 3.1.9.7 [32].

Samples were divided randomly into two main groups according to the contaminants into as shown in (Table 6) (Fig 6): Group (S): Saliva, group (D): Disclosing agent.

Each group was then subdivided into 4 subgroups (n=10) according to the surface cleansing method into: subgroup (C): Control group without cleansing, subgroup (V): Cleansing with Ivoclean agent, subgroup (A): Cleansing with air abrasion, subgroup (W): Cleansing with water.

Table 8: Sample grouping

Surface cleansing agent Surface Contaminant	Control group no cleansing (C)	Ivoclean (V)	Water (W)	Airborne particle abrasion (A)	Total
Group (S) Saliva	SC N=10	SV N=10	SW N=10	SA N=10	N=40
Group (D) Disclosing agent	SD N=10	DV N=10	DW N=10	DA N=10	N=40
Total	N=20	N=20	N=20	N=20	N=80

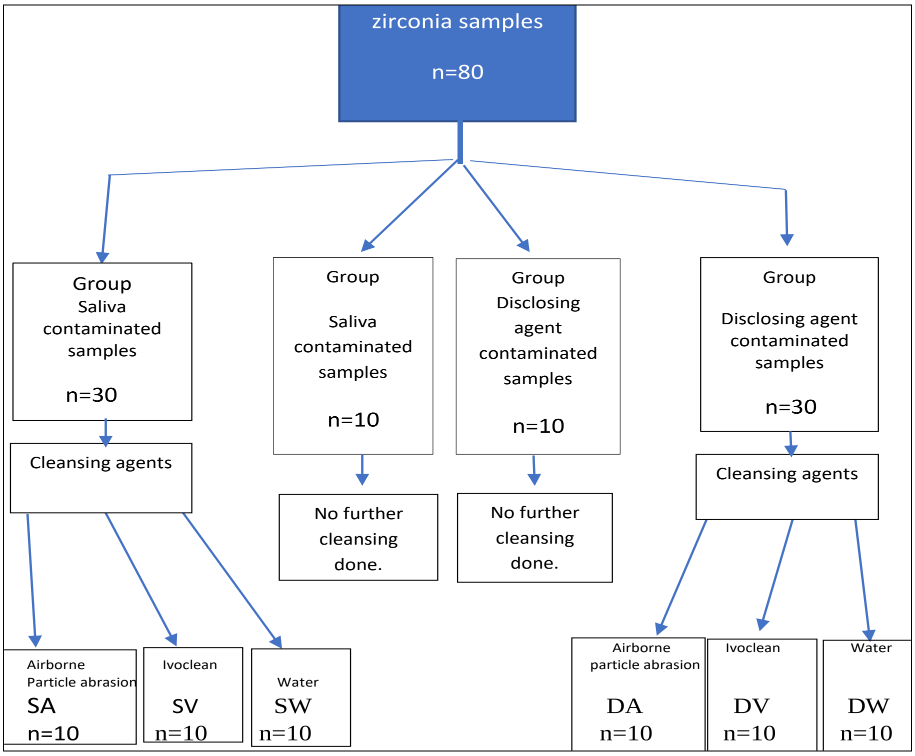

Fig 6: Illustrating sample grouping

Specimen preparation

A total of 2 blanks of zirconia were used in this research; each blank was cut using a low-speed diamond saw [5] (Fig 7) into 40 squares with total of 80 samples into dimensions of 12 x 12 x 2.4 mm (Fig 8) to be sintered to a final dimension of 10 x 10 x 2 mm (Fig 9). With each blank inserted, a new set of burs was used.

Fig 7: Sectioning of zirconia by Isomet

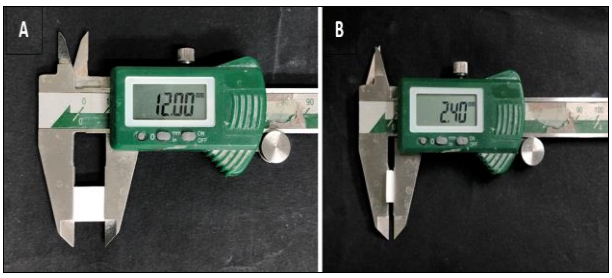

Fig 8: High translucent zirconia block dimensions before sintering (a) length of the square 12x12 mm (b) thickness of the square 2.4 mm

[5] IsoMet™ 4000 Linear Precision Saw, Buehler, USA

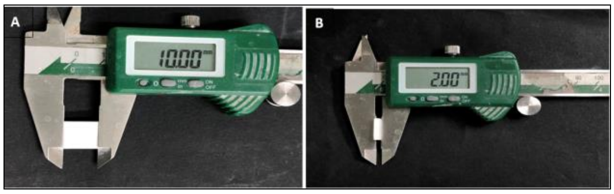

Fig 9: High translucent zirconia block dimensions after sintering (a) length of the square 10x10 mm (b) thickness of the square 2 mm

Zirconia sintering

All samples (n = 80) were sintered following the manufacturer's recommendations which provide sintering chart instructions using sintering furnace [6] (Fig 10).

Fig 10: Tabeo/M/Zirkon sintering furnace.

[6] TABEO/M/ZIRKON, MIHM-VOGT GMBH & CO. KG, Germany

The sintering furnace was programmed for a temperature increase from 20 °C to 1450 °C at a rate of 8 °C /minute, where the temperature remains constant for 120 minutes (Fig 11).

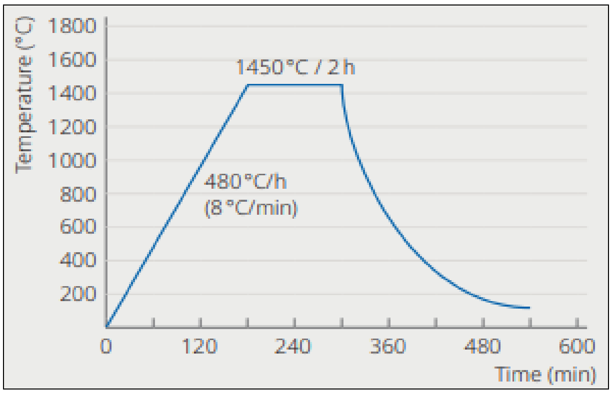

Fig 11: Sintering program

Difference in size after sintering of a sample already sintered and another one was yet to be sintered (Fig 12).
Due to shrinkage of zirconia, squares were cut to dimensions of 12 x 12 x 2.4 mm to obtain the required dimensions for this study of 10 x 10 x 2 mm, the calculation of the dimensional shrinkage was done according to the manufacturer instructions which is S: 18, 93%.

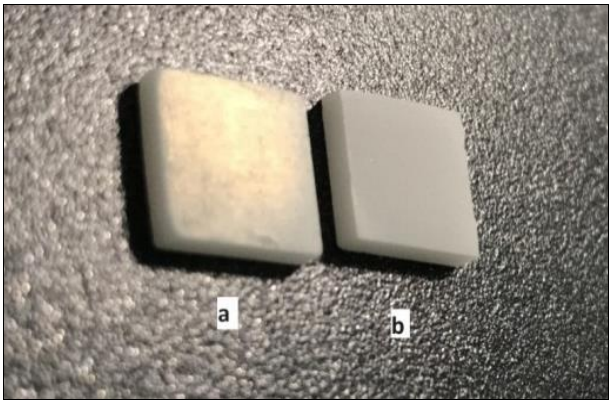

Fig 12: (a) size before sintering 12 x 12 x 2.4 mm and (b) size after sintering 10 x 10 x 2 mm

For ease of handling of the blocks a cylindrical mold (10 mm height and 25 mm an internal diameter) was designed using standard polypropylene pipes. Each specimen was embedded in mold which was filled with cold cure acrylic resin. The mold assembly was placed between two glass slabs, one below and one above to ensure that the outer surface of the zirconia disc was within the same level of the acrylic block and the mold (Fig 13).

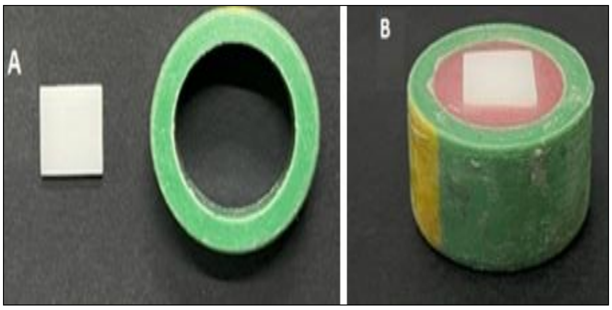

Fig 13: (A) mold to shape the block and (B) mold with cold cured acrylic resin and specimen in the middle of the mold

Contamination and cleansing of specimens

The same operator carried out all the procedures in this *in vitro* study according to the manufacturer's recommendation. The 80 specimens were air abraded with 50 µm aluminum oxide particles at 2.5 bar for 10s at a fixed distance of 10 mm which is recommended for zirconia, and then cleansed in an ultrasonic bath of distilled water for 5 minutes to remove any residues on the surface. Then, specimens were air dried to remove any liquid on the surfaces with oil-free compressed air for 10 seconds.
For the group of samples contaminated with fresh saliva, after air abrasion, fresh saliva collected from the author's own saliva who refrained from eating and drinking 2h before the collection procedure was applied and rubbed with a micro brush on the surface of zirconia samples and left for 2 minutes.
For the group of samples contaminated with the disclosing agent, a dual color disclosing solution was applied using a micro brush on the surface of zirconia samples of this group, then left for 2 minutes (Fig 14).

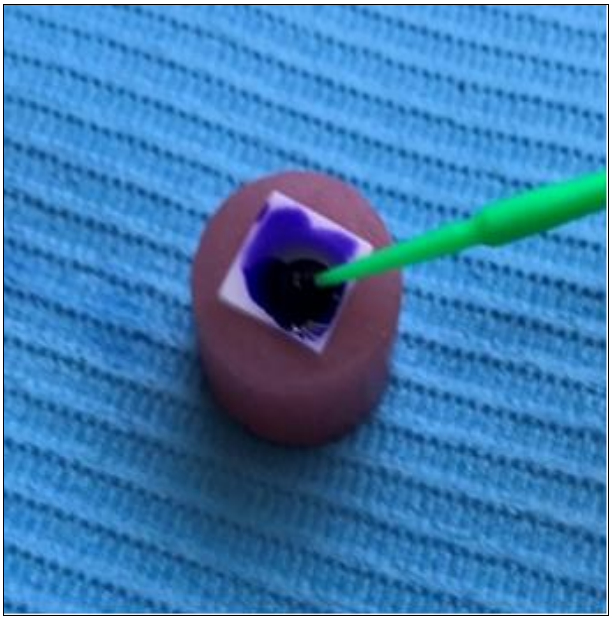

Fig 14: Sample contaminated with disclosing agent and rubbed with a micro brush.

After specimens were contaminated, contaminants were removed; for saliva and disclosing agent, the contaminated surface of specimens was rinsed with water for 20 seconds and dried with oil-free air for 30 seconds, while for the control group only dried with oil-free air for 30 seconds, then each group was further subdivided into four subgroups according to the cleansing method. No further cleansing was done for the control specimens contaminated with saliva and the disclosing agent, for specimens SV and DV, Ivoclean was used to clean the surface of the specimens: A drop of Ivoclean was dispensed over the surface of each block of the contaminated specimens. The solution was agitated with a micro brush and left for 20 seconds before washing, as recommended by the manufacturer (Figure 15). The surface was water rinsed until the color of the cleaning solution disappeared and dried with oil-free air, for specimens SA and DA, air particle abrasion was used to clean the surface of specimens at 2.5-bar pressure for 10 seconds at a 10-mm distance, 50 µm Al_2O_3, for specimens SW and DW, just water was used to clean the surface of the specimens for 10 seconds and then air dried with oil-free air.

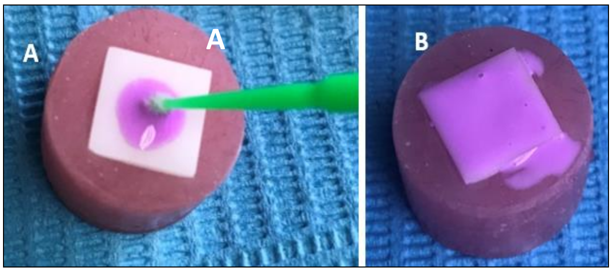

Fig 15: (a) Ivoclean dispensed on specimen surface by micro brush, (b) Ivoclean left for 20 seconds.

One sample from each group was tested for chemical element analysis using EDX (Energy Dispersive X-ray). EDX is an X-ray technique used to identify the elemental composition of materials because of its excellent surface sensitivity and suitability for examining and identifying surface contamination. EDX systems are attachments to (Scanning Electron Microscopy (SEM) instruments where the imaging capability of the microscope identifies the specimen of interest. The surface and structural morphology of the prepared samples were characterized using high-resolution scanning electron microscopy (SEM), and analysis experiments were carried out on a special instrument [7] (Figure 16). The diagram is an example of the readings obtained from the instrument after airborne particle abrasion cleansing of saliva contaminated HTZ surface. (Fig 17)

Fig 16: Universal instruments used in this study to identify the elemental composition of materials

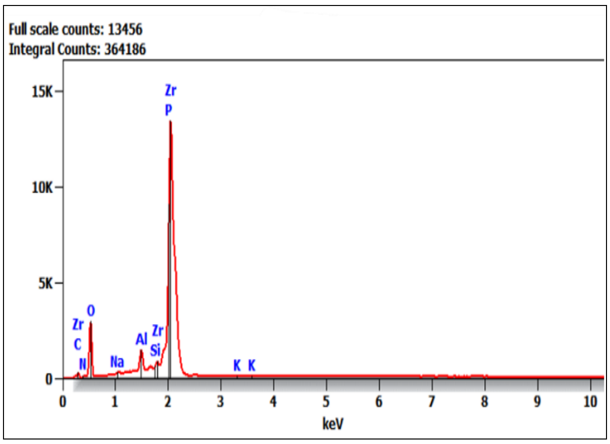

Fig 17: Diagram pre-sentative of one sample as example showing elements found after cleansing of contaminated zirconia surface with airborne particle abrasion

[7] FEI Quanta FEG 250 instrument, ELECMI, Madrid, Spain.

From the graph, we got some records showing the peaks for some elements. Then, data was collected in Table 7 in the results section to show which element is present to get an idea about the effect of contamination and cleansing on the elemental level.

Application of dual-cure resin to the surface

The resin cement was applied to all specimens by injecting it through a silicone mold (Ryle's tube) with an internal diameter of 3 mm and cut to a height of 3 mm (Figure 18) using a dual cure resin cement syringe with auto-mix tip, after each specimen cement material was polymerized using a light source of power 850 W/cm and intensity of 1,200-1,500 mW/cm^2 LED curing device [8] (Fig 19). Each specimen was cured for 20 seconds from a 10 mm distance. The mold was removed using blade no 11 from around the dual cure resin cement (Fig 20, 21 and 22).

Fig 18: Silicone mold (Ryle's tube) after cutting to the desired length

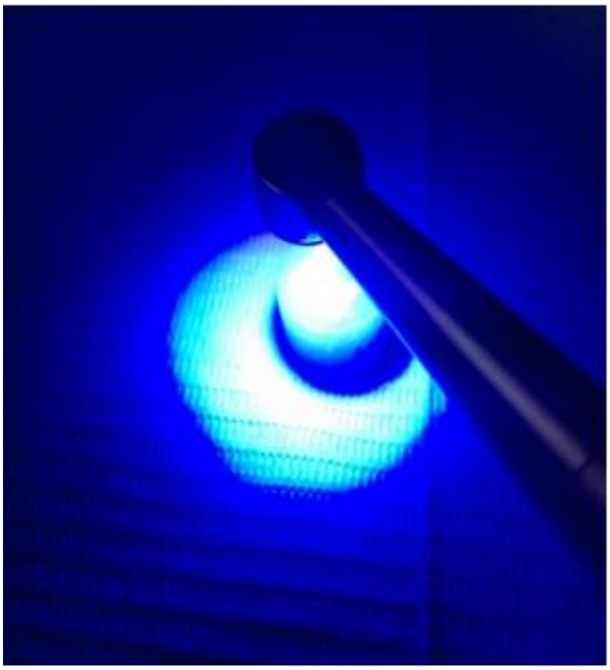

Fig 19: Curing resin cement from 10 mm distance

[8] curing pen by Eighteeth, Changzhou Sifary Medical Technology Co., Ltd., founded in Changzhou city, Jiangsu Province, China

Fig 20: Scalpel blade number 11 used to remove silicone mold from around the resin cement

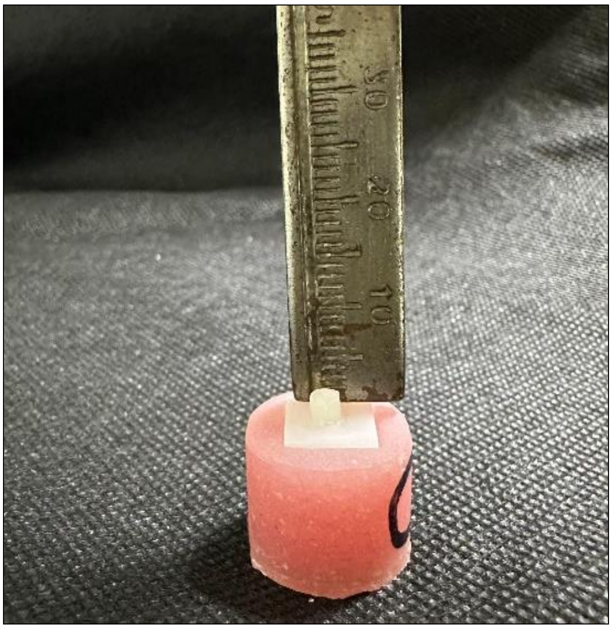

Fig 21: Dual cure resin cement on the specimen after curing and removal of the silicone mold

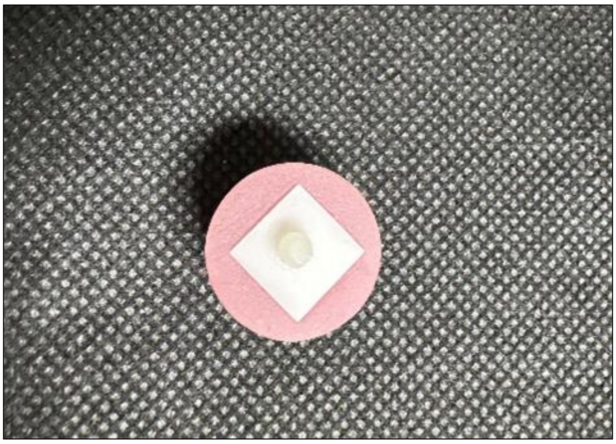

Fig 22: Specimen after resin cement bonding and removal of silicone mold

All specimens were thermocycled 5000 cycles cold water bath immersion for 30 seconds at 5 °C. Hot water bath immersion for 30 seconds at 55 °C [9]. Dwell Time 10 seconds before resin bond testing. (Fig 23).

Fig 23: Thermocycler used for artificial ageing of specimens in this study.

All the specimens were subjected to a shear bond strength test (SBS) using a universal testing machine [10]. Each acrylic block containing the square specimen was mounted on the lower jig of the universal testing machine. Screws were tightened until the acrylic block was properly supported and fixed between the upper and lower jigs. The samples were positioned parallel to the loading direction of the jig of the universal testing machine (Figure 24). The following formula was used to calculate SBS data: fracture load/bonding surface area (inner diameter) = N/mm² = MPa.

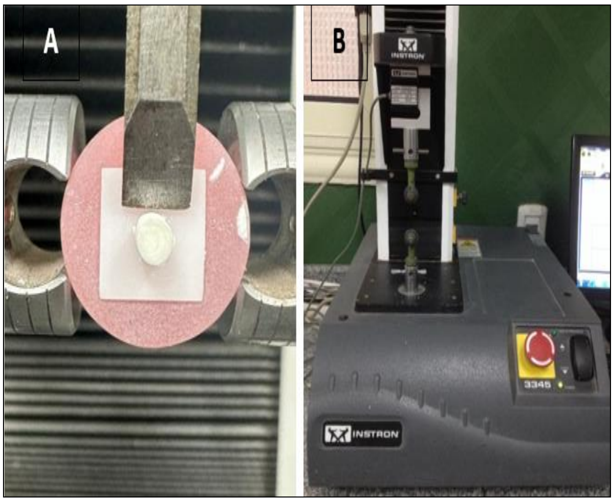

Fig 24: (A) Shear bond strength test, (B) Universal testing machine

Failure mode analysis

Following fracture, the interfaces of the specimens were meticulously analyzed using a digital camera to categorize the failure modes. Debonded surfaces were classified as exhibiting cohesive failure within the luting resin, adhesive failure at the ceramic-cement interface, or a combination of both (Mixed adhesive/cohesive). (Fig 25) The extent of each failure mode (Cohesive, adhesive, mixed) was quantified for each specimen by calculating the area affected and expressing it as a percentage of the total bonding surface area within each test group [33].

[9] Thermocycler, SD Mechatronic, Feldkirchen-Westerham, Germany
[10] Instron model 3345 universal testing machine, University Ave Norwood, MA, US

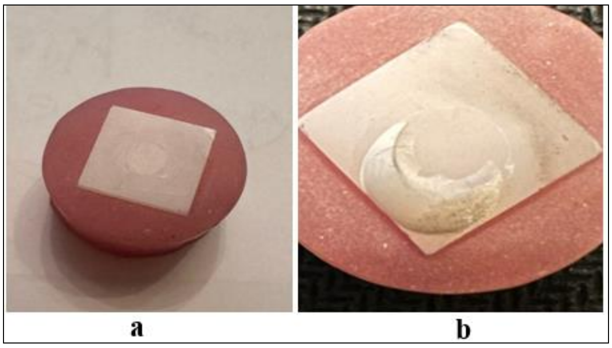

Fig 25: (a) cohesive failure and (b) mixed failure

Statistical analysis

To analyze different groups based on categorical data (frequency, percentage), a chi-square test was used, followed by pairwise comparisons using z-tests with Bonferroni correction. For numerical data (mean, SD), normality was confirmed with the Shapiro-Wilk test and then analyzed by two-way ANOVA and Tukey's post hoc test. Further comparison of specific effects was conducted using Bonferroni correction with the combined error term from the main ANOVA model. All tests had a significance level of $p<0.05$ and all the statistical analysis were conducted using R statistical analysis software version 4.3.0 for Windows [11].

Results

EDX

This study's EDX results (Table 7) showed that after saliva immersion and applying a disclosing agent, an organic coating of C, O, and Si on the ceramic surface was associated with a considerable drop in ceramic bond strength.

Table 9: Percentage values by weight percent of the elements detected on specimen surfaces after contamination and cleaning protocols.

	Groups							
	S	D	SA	SW	SV	DA	DW	DV
Zr	63.15	64.28	65.48	61.52	66.35	66.42	62.06	64.79
C	2.83	2.62	2.30	2.84	1.88	2.44	5.16	2.24
Si	0.05	0.00	0.00	0.00	0.02	0.00	0.00	0.00
Al	1.51	1.36	1.42	1.27	1.23	1.25	0.98	1.22
K	0.04	0.00	0.00	0.10	0.00	0.06	0.00	0.09
P	2.39	2.17	1.37	0.57	1.65	2.28	0.43	0.00
O	22.70	22.35	20.84	21.53	20.89	21.65	22.04	20.67

Abbreviations: Zr, zirconium; C, carbon; Si, silicon; Al, aluminum; K, potassium; P, phosphate; S, saliva; D, disclosing agent; O, oxygen; SA, saliva contaminated samples cleaned with airborne particle abrasion; SV, saliva contaminated samples cleansed with Ivoclean universal cleaning agent; SW, saliva contaminated samples cleansed with water; DA, disclosing agent contaminated samples cleansed with airborne particle abrasion; DV, disclosing agent contaminated samples cleansed with Ivoclean universal cleaning agent; DW, disclosing agent contaminated samples cleansed with water.

One sample from each group was examined for EDX. Table 7 details the quantitative measurements of elements identified by EDX analysis on the specimen surfaces. Zirconium (Zr), oxygen (O), and carbon (C) were the most prevalent elements detected consecutively. Notably, airborne particle abrasion yielded the highest level of Zr, while the control group exhibited the highest level of O.

II-Shear Bond strength

1. Effect of different variables and their interaction on SBS of dual cure resin cement to on HTZ

Effect of different variables (saliva and disclosing agent) and their interaction on bond strength (MPa) are presented in table (8).

There was a significant interaction between contamination and cleaning methods ($p<0.001$).

Interactions

Mean and standard deviation (SD) values of bond strength (MPa) for different contamination methods and cleaning methods are presented in Table (9).

A-Effect of cleansing method on SBS of dual-cure resin cement to on HTZ

Following Saliva contamination

There was a significant difference between different groups ($p<0.001$). The highest value was found in samples treated with air abrasion (32.25±2.00), followed by Ivoclean (18.28±0.64), then these cleansed with water (9.13±0.31), while the lowest value was found in the control group (9.04±0.20). Post hoc pairwise comparisons showed samples cleansed with air-abrasion samples to have significantly the highest values than other groups ($p<0.001$). In addition, they showed Ivoclean cleansed samples to have a significantly higher value than samples treated with water and untreated samples ($p<0.001$).

Following Disclosing agent contamination

There was a significant difference between different groups ($p<0.001$). The highest value was found in samples cleansed with air abrasion (30.45±1.68), followed by Ivoclean (19.28±0.57), then the control group (9.47±0.46), while the lowest value was found in water-cleansed samples (9.21±0.84). Post hoc pairwise comparisons showed air-abraded samples to significantly have the highest values than other groups ($p<0.001$). In addition, they showed Ivoclean to have a significantly higher value than samples treated with water and untreated samples ($p<0.001$).

B-Effect of cleansing method on SBS of dual-cure resin cement to HTZ

Control

There was no statistically significant difference between control group samples contaminated with saliva and samples contaminated with disclosing agent. (p=0.358).

Water

There was no statistically significant difference between contaminated samples cleansed by water. (p=0.860).

Air abrasion

Saliva-contaminated samples (32.25±2.00) had a significantly higher SBS value than samples contaminated by the disclosing agent (30.45±1.68) ($p<0.001$).

[11] R Core Team (2023). R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. URL https://www.R-project.org/.

Table 10: Effect of different variables and their interactions on bond strength (MPa) (µm)

Source	Sum of squares	DF	Mean square	f-value	p-value
Contamination method	0.10	1	0.10	0.10	0.757ns
Cleaning method	6605.34	3	2201.78	2040.26	<0.001*
Contamination method * Cleaning method	22.05	3	7.35	6.81	<0.001*

DF = Degree of freedom*; significant ($p \leq 0.05$) ns; non-significant ($p>0.05$)

Ivoclean

Disclosing agent contaminated samples (19.28±0.57) had a significantly higher SBS value than saliva contaminated samples (18.28±0.64). (p=0.035).

Table 11: Comparisons of bond strength (MPa) for different contamination methods and cleaning methods

Contamination method	Bond strength (MPa) (mean ±SD)				p-value
	Control	Water cleaning	Air abrasion cleaning	Ivoclean cleaning	
Saliva	9.04 ± 0.20^{C}	9.13 ± 0.31^{C}	32.25 ± 2.00^{A}	18.28 ± 0.64^{B}	<0.001*
Disclosing agent	9.47 ± 0.46^{C}	9.21 ± 0.84^{C}	30.45 ± 1.68^{A}	19.28 ± 0.57^{B}	<0.001*
p-value	0.358ns	0.860ns	<0.001*	0.035*	

Means with different superscript letters within the same horizontal row are significantly different *; significant ($p \leq 0.05$) ns; non-significant ($p>0.05$).

III-Mode of failure

Intergroup comparisons, frequencies, and percentages of modes of failure in different contamination and cleansing methods are presented in Table (10).

A-Effect of contamination method on mode of failure of dual-cure resin cement to HTZ

Saliva

There was a significant difference between the different groups, with a significantly higher percentage of the control group and water-treated samples having adhesive failures, and a higher percentage of air-abraded and Ivoclean-treated samples having mixed failures ($p<0.001$).

Disclosing agent

There was a significant difference between different groups (p=0.002). Samples of control and water-treated groups had a significantly higher percentage of adhesive failures than air-abraded samples and a higher percentage of air-abraded samples having mixed failures than untreated and water-treated samples.

B. Effect of cleaning method on mode of failure of dual-cure resin cement to HTZ

The cleaning method had no statistically significant effect on mode of failure within all treatments ($p>0.05$).

Table 12: Frequencies and percentages of mode of failure in different contamination methods and cleaning methods

Contamination	Failure mode	Control		Water		Air abrasion		Ivoclean		p-value
		N	%	n	%	N	%	N	%	
Saliva contaminant	Adhesive	10^{A}	100.0%	10^{A}	100.0%	2^{B}	20.0%	4^{B}	40.0%	<0.001*
	Cohesive	0^{A}	0.0%	0^{A}	0.0%	0^{A}	0.0%	0^{A}	0.0%	
	Mixed	0^{A}	0.0%	0^{A}	0.0%	8^{B}	80.0%	6^{B}	60.0%	
Disclosing agent contaminant	Adhesive	10^{A}	100.0%	8^{A}	80.0%	2^{B}	20.0%	6^{AB}	60.0%	0.002*
	Cohesive	0^{A}	0.0%	0^{A}	0.0%	0^{A}	0.0%	0^{A}	0.0%	
	Mixed	0^{A}	0.0%	2^{A}	20.0%	8^{B}	80.0%	4^{AB}	40.0%	
p-value		NA		0.136ns		1ns		0.371ns		

NA: Not Applicable, Means with different superscript letters within the same horizontal row are significantly different *; significant ($p \leq 0.05$) ns; non-significant ($p>0.05$)

Discussion

Modern dentistry widely uses zirconia-based restorations due to their exceptional biocompatibility, strength, and aesthetically pleasing qualities [34]. In the era of minimally invasive dentistry, a strong, long-lasting bond to zirconia is preferred when adhesion is the primary means of restoration retention or when enhancing the mechanical characteristics of the tooth prosthesis complex is required [35]. The zirconia's surface is hard to etch and inert. The restorations can withstand severe chemical difficulties in the oral environment thanks to this feature. But when it comes to zirconia bonding, this becomes a big problem [36-40]. Sandblasting with alumina microparticles or using cements based on resins containing phosphate monomers can produce an ideal bond to zirconia [36].

During the intraoral try-in step, it is inevitable that saliva, blood, or disclosing agent would contaminate the pre-treated restoration surface. Disclosing agent is used to check the fitting surface of restoration during try in to check if there is ill-fitting area to be adjusted. Contamination makes bonding methods more technique-sensitive and increases the difficulty of achieving good retention. Previous studies revealed that when dental enamel was exposed to saliva for longer than a second, a tenacious pellicle formed on the enamel that could not be removed with a water rinse [41]. Bacteria, inorganic particles, and proteinaceous materials can adhere to the zirconia surface in saliva and blood, negatively impacting resin bonding [19, 20].

This study aimed to identify the optimal cleansing method for maximizing shear bond strength of resin cement to zirconia restorations contaminated with saliva and disclosing agent. Additionally, it sought to evaluate the efficacy of various cleansing agents in mitigating the detrimental effects of contamination on bond strength.

A cylindrical mold (10 mm height and 25 mm internal diameter) was used in this study to make an acrylic base for the specimens for ease of handling. In this study, the airborne particle abrasion surface treatment strategy was chosen to improve the bond strength of zirconia. Nonetheless, this method is regarded as the gold standard for zirconia treatment and is frequently cited in research aimed at enhancing surface area and roughness for bonding via a micromechanical bonding mechanism [10].

The study used 50 µm sand particles under 2.5 bar pressure, 10 mm apart, and 10 seconds at a perpendicular angle as the air abrasion conditions [42]. These parameters are based on earlier research. Moon *et al*. (2016) [21] found that the highest shear bond strength between zirconia and resin cement was achieved by sandblasting with 50 µm particles at 4 bar for 20 seconds at 45° or 90°. Other studies, however, indicated that in order to prevent surface damage, zirconia should be sandblasted at low pressure (1 to 2 bars) and with small powder particle size (>50 µm) [22, 23]. In this study, the spacing has been changed from 25 mm to 10 mm because Zeighami *et al*. (2017) [43] found that increasing the distance to 25 mm decreases surface roughness significantly and, as a result, the zirconia bonding technique.

To standardize the effect of sandblasting for all the tested specimens in the present study, an endodontic ruler was used to ensure the distance was 10 mm and stable. The nozzle of the sandblaster, ruler and specimen were all properly stabilized on the base of the sandblasting unit.

With the advent of ceramic cleaners, clinicians now have an easy-to-follow cleansing procedure for recovering ceramic surfaces following contamination. Ivoclean is marketed by the manufacturer as an alkaline extraoral universal ceramic cleaner. Ivoclean works well for restoring ceramic surfaces [25, 28]. Zirconia oxide particles that are highly concentrated and form a concentration gradient in Ivoclean give the solution a higher affinity for phosphate than the ceramic surface. Organic contaminants are eliminated from the zirconia surface by increased affinity in the solution, which can subsequently be washed off with water [25, 28].

The contaminants used in this study were selected because they are either clinically used throughout the try-in procedure for any prosthetic restoration or clinically present in the oral environment. The impact of specific contaminants on the bond strength of the ceramic material to be cemented was examined in some previous articles. It has been demonstrated that surface contamination with silicone-disclosing agent and saliva during the intraoral try-in weakens the bond strength of the restorations [2].

In this *in vitro* study two contaminants were used, fresh saliva and disclosing agent. Saliva is made up of organic components such salivary proteins, microorganisms, and food particles dissolved in water. Salivary proteins would bind to surfaces other than the tooth after immersion [45]. But also on various other materials of restorations [46, 47].

Self-adhesive dual cure universal resin cement was the cement used in this study. Since it is a self-adhesive resin cement, it saves time in clinical practice as no need for etching and bonding of the tooth surface, and it is considered a strong resin cement and moisture tolerant. Previous studies proved that cements with a high degree of conversion are expected to provide good mechanical properties. The concentration of the monomer and catalyst and the ambient temperature influence the degree of conversion of auto-polymerized cement [44].

Intraoral try-ins inherently expose restorations to saliva contamination, posing a challenge for subsequent adhesive cementation. Confirming this concern, Chung *et al*. in (2009) [48], demonstrated that saliva significantly compromises the bond quality of resin cements, also another study made by Kawaguchi-Uemura *et al*. in (2018) [49] proved that the long-term durability of bonds between CAD/CAM resin blocks and luting agent cement was significantly reduced by saliva contamination. Another study made by Pitta *et al*. in 2018 [50] proved that in general, saliva contamination and aging decreased bonding efficacy. In this study fresh saliva was used which was obtained from the author himself after he avoided eating and drinking for two hours before obtaining saliva. Ionic bonds are created when salivary organic material comes in contact with the zirconia surface. Therefore, water alone cannot interact with or dissolve salivary proteins to decontaminate the zirconia surface. The technique of saliva application was made according to previous studies, spreading saliva over specimen with a micro brush then left undisturbed for 1 minute.

Previous study made by Szep *et al*. in 2003 [51] used the disclosing material as contaminant and its effect on the bond strength of ceramic material to composite was assessed, Statistical analysis revealed no significant decrease in shear bond strength between ceramic and composite cement when silicone disclosing procedures were employed prior to surface conditioning.

The cleansing methods used in this study were chosen according to their availability in the clinic as a chair-side procedure so it will be an easy procedure and not time consuming, they were chosen also due to their presence in literature as cleansing methods used to clean the contamination off the surface of the restoration to be cemented, former research has documented the implementation of diverse cleaning techniques, encompassing both mechanical and chemical approaches, to enhance the bond strength of contaminated surfaces [9, 52]. This study employed airborne particle abrasion using 50 µm aluminum oxide (Al_2O_3) particles at a pressure of 2.5 bar for 10 seconds and a working distance of 10 mm. Based on the observed improvement in bond strength between luting resin and zirconia ceramic, this protocol is recommended as a standardized surface treatment for achieving optimal clinical outcomes.

Our EDX findings align with those of Wattanasirmkit and Charasseangpaisarn (2019) [27] by demonstrating that no cleansing method achieved complete surface decontamination. While sandblasting yielded the most favorable surface element ratios, suggesting superior cleaning capabilities, residual contaminants were still detectable.

The detection of carbon (C) element on the zirconia surfaces, likely residual contamination from saliva, indicates the inherent limitations of achieving complete surface cleanliness. Among the tested methods, water cleaning exhibited the highest C content, followed by Ivoclean and then airborne particle abrasion, mirroring the bond strength results for the cleaning methods used. Besides, EDX analysis revealed zirconium and oxygen as the predominant elements on the zirconia surfaces, consistent with the expected composition. However, the detection of phosphorus, a key component of salivary phospholipids, was inconsistent across all cleaning groups. This lack of clarity could be attributed to two possibilities: either the amount of phosphorus present was exceedingly low compared to the investigated surface area, or complete removal of this element may have been achieved in certain groups. This result diverged from those of Phark *et al*. [20], who observed a notable increase in surface phosphorus content on zirconia specimens cleaned with phosphoric acid compared to other methods.

While EDX analysis provided qualitative insights into the elemental composition of surface contaminants by quantifying their relative volumetric percentages, it lacked the sensitivity to quantify element abundance or differentiate between contaminant distribution as thin films or clusters. To further elucidate contaminant-related failure mechanisms in future studies, techniques like X-ray photoelectron spectroscopy (XPS) would be invaluable for their superior elemental depth profiling and chemical state characterization capabilities.

In the present study, an aging protocol of a 5000 thermocycle was done. The duration and application of the thermal aging protocol may affect the bond strength and failure type [8, 53, 54]. Thermocycling is used as an aging method to simulate intra-oral situations. The majority of studies showed that thermocycling significantly reduce the bond strength [53, 54]. On the other hand, thermal cycling may not have a significant effect on the bond strength but it can lead to spontaneous de-bonding of the specimen [55, 56]. According to the ISO norm 10477, the minimum number of thermocycles was proposed as 5000 to assess metal resin bonds [57]. In the present study thermocycling was used for 5000 cycles at cold water bath immersion for 30 seconds at 5 °C. Hot water batch immersion for 30 seconds with a dwell time of 10 seconds which is equivalent to six months intraoral.

In this study, the SBS test method was used, but it does not result in a uniform distribution of stress on the specimens' surfaces [58]. Since the micro tensile test method obtains more accurate and controlled data, it may provide more reliable results [59].

This study rejected the null hypothesis because the SBS of contaminated zirconium was statistically different between different surface cleansing methods and adhesive resin cement. This aligns with established literature (Irmak *et al*., 2018; Noronha *et al*., 2020; Phark *et al*., 2009; Yoshida, 2018) [60, 26, 20, 5], emphasizing the crucial role of thorough surface cleaning to achieve durable bond strength on contaminated zirconia.

In agreement with our results contaminants' occupation of the oxide layer on the zirconia surface, which restricts the bonding agent's connectivity, is the reason for the bond failure seen in the control groups with no cleansing agent (According to Angkasith *et al*. 2016, Kim *et al*. 2015, Wattanasirmkit & Charasseangpaisarn 2019, and Wille *et al*. 2015) [25, 28, 27, 61], In some earlier articles, it was discussed how to clean contaminants from ceramic surfaces before cementing them in order to strengthen the bond between the surfaces and the restorative materials, as drying contaminated surfaces only yielded the weakest bond between any contaminant and any restorative material. Previous investigations demonstrated that the cleaning solutions could partially clean the silicone-disclosing medium and the saliva-contaminated surface [3, 20].

In agreement to our results of shear bond strength of (32 MPa) the airborne particle abrasion of the zirconia-contaminated surface increases the bond strength of the resin cement by completely cleaning the surface and creating a more microporous surface (Russo *et al*., 2019; Tunc *et al*., 2016; Wegner, 2000; Yang *et al*., 2007; Yoshida, 2018) [62-64, 19, 5].

Air-borne abrasion can significantly increase bond strength between resin cement and zirconia through various mechanisms; it utilizes a stream of compressed air carrying abrasive particles, typically aluminum oxide, to roughen the zirconia surface [11-18]. This process increases the surface area available for micromechanical interlocking between the resin cement and the zirconia. Imagine trying to glue two smooth surfaces together compared to two rough surfaces; the rougher surfaces provide more "grip" for the glue, resulting in a stronger bond. In addition, abrasion creates microscopic irregularities on the zirconia surface, enhancing the wettability of the resin cement. This allows the resin to flow more easily into the surface irregularities, promoting a more intimate contact and stronger bond. Because air abrasion raises surface roughness, cleans, removes impurities, improves wettability, and increases surface energy, it is used to achieve mechanical interlocking between two materials.

The airborne particle-abraded zirconia specimens had the greatest SBS values. The results show that aluminum oxide sandblasting considerably increases bond strength, as has been shown in other studies **(**Angkasith *et al*., 2016; Çakırbay Tanıs¸ *et al*., 2019; Kim *et al*., 2015; Tunc *et al*., 2016; Yang *et al*., 2007; Yoshida, 2018) [25, 65, 28, 63, 12, 5].

In this study, it was demonstrated that zirconia surfaces contaminated with both saliva and a silicone-disclosing medium exhibited significantly lower shear bond strength (SBS) upon bonding. This reduction in bond strength is likely attributable to the presence of residual organic matter from the contaminants, specifically organic substances from saliva and silicone particles from the disclosing medium, adhering to the zirconia surface [28, 1]. However, the SBS was improved, and it was not significantly different from a non-contaminated surface when the surfaces were cleansed with air particle abrasion and Ivoclean but not with water. This aligns with existing research suggesting that the cleansing agents could clean the organic substance on saliva-contaminated surfaces and partially clean the silicone-disclosing medium [20, 3].

According to several studies contradicting our results (Aladag *et al*., 2015; Feitosa *et al*., 2015; Wattanasirmkit & Charasseangpaisarn, 2019) [66, 24, 27], surface may have cracks and deformations because of sand blasting. In contrast to other studies that identified distortion, damage, and cracks (Aladag *et al*., 2015; Chintapalli, Marro, Jimenez-Pique, & Anglada, 2013; Feitosa *et al*., 2015) [66, 67, 24], there was no deformation because of SB was found in this study. This is thought to be due to the use of recommended pressure level and sand particle size within the limits for the zirconium material (Blatz, 2016; Skienhe, Habchi, Ounsi, Ferrari, & Salameh, 2018) [68, 69].

Considering both effectiveness and potential drawbacks, airborne particle abrasion will be adopted as the initial treatment for all specimens. While its inherent roughness may facilitate contaminant retention and complicate thorough cleaning, especially for the disclosing agent, the practical challenges of accessing and adequately decontaminating internal restoration surfaces, with their inherent lack of flatness compared to the experimental specimens, render this approach the most feasible first step.

Airborne particle abrasion offered a dual benefit: it eliminated contaminants known to disrupt chemical bonding [70, 71] and simultaneously removed the outermost ceramic layer mechanically, revealing a pristine surface ideally suited for strong bonding with the phosphate-modified adhesive monomer in the resin cement.

In agreement with our results Ivoclean can enhance bond strength (18.28 and 19.28 MPa) in comparison to the not treated and water cleansed group. Ivoclean, which combines alkaline and zirconium oxide particles, has the potential to significantly strengthen the bonding between zirconia and resin cement in several ways. Zirconium oxide particles are abrasive, removing debris, blood, and saliva from surfaces [30]. The alkaline further improves cleansing performance through its capacity to dissolve organic contaminants and encourage surface activation. Additionally, the zirconium oxide particles in Ivoclean increase the surface area accessible for micromechanical interlocking between the zirconia and resin cement by causing microscopic irregularities on the zirconia surface. The resin can enter irregularities thanks to its roughened surface, creating a stronger and more intimate bond [29].

Unlike in the previous groups, which were cleansed by airborne particle abrasion (SA and DA) or Ivoclean (SV and DV), the SBS was lowest in the two control groups (SC and DC) without a cleaning agent applied, and in the groups cleaned with water (SW and DW) with no statistically significant difference between them. This finding is further supported by the fact that all the specimens in these groups had adhesive failure types. The reason for the bond failure seen in the control groups is that contaminants limit the connection of the bonding agent by occupying the oxide layer on the zirconia surface (Angkasith *et al*., 2016; Kim *et al*., 2015; Wattanasirmkit & Charasseangpaisarn, 2019; Wille *et al*., 2015) [25, 28, 27, 61].

As for the mode of failure analysis, all control group samples (SC and DC) (100%) and subgroups cleaned with water (SW and DW)) (100%) failed adhesively. Those groups demonstrating predominately adhesive failure indicate weak unstable bonds to zirconia. Groups cleansed with airborne particle abrasion (SA and DA) and Ivoclean (SV and DV) showed mixed failures, predominantly cohesive failure, indicating a durable bond to zirconia. A strong correlation was observed between shear bond strength (SBS) and failure mode in each group. Notably, both groups exhibited cohesive failure within the adhesive resin and mixed failures, indicating high bond strength between zirconia and adhesive resin, suggesting resistance to contamination by saliva and disclosing medium. This potentially suggests that mechanical retention, facilitated by airborne particle abrasion, played a more prominent role in bond strength compared to chemical retention, which remained irreplaceable. Nonetheless, evaluating the long-term influence of leakage and aging on zirconia-resin bonding durability through dedicated studies is crucial.

Some studies suggested that a bond strength of 15 to 21 MPa is necessary for clinical use [72]. In this study, the mean shear bond strength values of zirconia surface contaminated with saliva and cleansed by airborne particle abrasion is 32.25 MPa or Ivoclean is 18.28 MPa, while the mean shear bond strength values of zirconia surface contaminated with disclosing agent and cleaned by airborne particle abrasion is 30.45 MPa or Ivoclean is 19.28 MPa, which were considered to be clinically acceptable, while the mean shear bond strength values of high translucent zirconia surface contaminated by saliva and disclosing agent that were not cleansed or cleansed with water were 9.04 MPa, 9.47 MPa, 9.13 MPa and 9.21 MPa respectively, which were clinically not within the acceptable range.

Conclusions

This study identified several key conclusions within its limitations. Firstly, saliva and disclosing agents were confirmed to negatively impact the shear bond strength (SBS) of resin cement to zirconia surfaces. Secondly, different cleansing methods displayed varying efficacy in removing contaminants and achieving optimal bond strength comparable to uncontaminated zirconia. Notably, airborne particle abrasion emerged as the most effective method, effectively cleansing contaminated surfaces and significantly enhancing SBS. Although all tested zirconia samples, regardless of contamination and cleaning, ultimately exceeded the minimum acceptable SBS range for clinical use (15-21 MPa), significantly lower values were observed in non-cleansed and water-cleansed groups, highlighting the essential role of thorough cleansing in maximizing bond strength.

Conflict of Interest

Not available

Financial Support

Not available

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