

INFLUENCE OF SALIVA CONTAMINATION ON RESIN BOND DURABILITY
TO ZIRCONIA – EFFECT OF CLEANING METHODS

by

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This thesis is dedicated to the love of my life, my son Hrian; to my beloved husband, Harshil; and to my parents and my family.

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INTRODUCTION

Restorations made of porcelain fused to metal (PFM) are stable and reliable for oral rehabilitation for missing teeth. They have relatively high mechanical strength, marginal integrity, and rare negative response to high precious metal. However, there is always a concern about hypersensitivity reaction to certain base metal materials, such as nickel or cobalt. Moreover, both the opacity and the dark appearance of metal base structures have a negative influence on the esthetic results of the prosthesis. Given the demand for esthetic or natural appearance and better biocompatibility, the application of a metal-free dental prosthesis has growing promise as an alternative to the use of PFM restorations.

Dental ceramics are very popular as highly esthetic restorative materials that better simulate natural dentition. Other desirable characteristics of dental ceramics include translucency, fluorescence, chemical stability, biocompatibility, high compressive strength, and a co-efficient of thermal expansion similar to that of tooth structure.¹ The introduction of zirconia into modern dental practice has greatly advanced the development of metal-free dentistry. Currently, various types of zirconia ceramics are being used extensively to fabricate dental restorations owing to high fracture toughness and aesthetic properties.²⁻⁵ In terms of fracture resistance, zirconia-based fixed partial dentures (FPDs) have the potential to withstand physiologic forces of occlusion in the posterior region and therefore provide an interesting alternative to metal ceramic restorations.⁶ Moreover due to its excellent biocompatibility and chemical stability, zirconia has been utilized in fabrication of dental implants and abutments. It has now

gained more attention from dentists and researchers. However, the application of a zirconia-based restoration is constrained by its chemical inertness and the resultant relative weak bonding properties, including resin to zirconia, and porcelain to zirconia bonding. Therefore, many investigations are carried out to improve zirconia's bonding ability. Typically, resin cements are used for luting zirconia crowns or frameworks to the tooth abutments.⁷⁻¹⁵ However, a clinical problem with the use of zirconia restorations is the difficulty in achieving a reliable and durable bond between the resin luting agent and the ceramic.^{2,7,8,12-20} A strong resin bond relies on chemical adhesion and/or micromechanical interlocking created by surface conditioning methods such as roughening. Current roughening techniques consist of grinding, abrasion with diamond rotary instrument, airborne particle abrasion with alumina or silica-modified alumina particles, acid etching, or a combination of these techniques.²¹⁻²⁴

The composition and physical properties of zirconia differs from conventional glass-based ceramics. Zirconia is densely sintered and does not contain a glassy phase; therefore, it cannot be etched with hydrofluoric (HF) acid to create a micro-retentive etching pattern. Thus, in order to achieve a reliable and durable bond in a wide range of clinical applications, alternative bonding strategies are required.

Meanwhile, another major issue pertaining to bonding of ceramic restorations is related to its potential contamination before cementation. After sandblasting and clinical try-in procedures, zirconia can get contaminated with saliva and/or blood. As with many metals, zirconium shows a strong affinity towards the phosphate group found in saliva and other fluids, which reacts with the zirconia surface and makes bonding difficult.

Recently, a new cleaning agent called Ivoclean[®] (Ivoclar-Vivadent, Schaan, Liechtenstein), which is an alkaline suspension of zirconium oxide particles, was developed to remove the contamination from zirconia in an effort to improve bonding to resin cements. Due to its size and the concentration of the particles in the medium, phosphate contaminants are much more likely to bond to them than to the surface of the ceramic restorations. Ivoclean adsorbs the phosphate contaminants preferentially, thus leaving behind a clean zirconium oxide surface.

PURPOSE

The purpose of this study was to evaluate the influence of saliva contamination and the effect of several cleaning methods, on the resin bond durability to zirconia. Shear tests were performed to assess the shear bond strength of specimens after 24 h of storage or after thermocycling as an aging method.

Null Hypothesis

The null hypothesis to be tested is that cleaning methods or storage conditions will not influence bonding to zirconia.

Alternative Hypothesis

The cleaning methods employed after saliva contamination will positively influence bonding to zirconia. More specifically, the shear bond strength of resin cement to zirconia was improved after cleaning with Ivoclean both immediately and after thermal aging (thermocycling).

REVIEW OF LITERATURE

INTRODUCTION TO ZIRCONIA

Porcelain fused to metal restoration was introduced into dental clinical practice more than 50 years ago and became very popular in the fabrication of fixed dental restorations due to their strength, marginal integrity, and durability. However, there is a concern about the reported incidence of hypersensitivity reaction to certain metals used in the fabrication of PFM restorations like nickel and cobalt. Also, the dark appearance of metal base structures has a detrimental effect on the final esthetic of dental prostheses. Therefore, there is growing interest in the development of a metal-free, naturally appearing, biocompatible dental prosthesis as an alternative to metal ceramic restorations.

In search of the best restorative material, all ceramic systems were considered as the best option. Dental ceramics can be classified in various ways depending on their properties. Chemically, dental ceramics can be divided into following categories depending on its core material: Glass ceramics (lithium-disilicate, leucite, feldspathic), alumina (aluminum-oxide), zirconia (Yttrium tetragonal zirconia polycrystals).

In recent years, zirconia has become very popular due to its favorable esthetic properties, mechanical properties, and biocompatibility.² The fracture toughness of densely sintered zirconia ceramics is more than 1000MPa. The first biomedical application of zirconia occurred in 1969 but its use in dentistry started in the early 1990s. Zirconia ceramics are currently used for fixed restorations as a framework material due to their mechanical and optical properties. In terms of fracture resistance, zirconia-based fixed partial dentures have the potential to withstand physiological occlusal forces

applied on the teeth and therefore provide an interesting alternative to metal-ceramic restorations. Zirconia ceramics have been used in the fabrication of ceramic veneers, single crowns, inlays and onlays, fixed partial denture prosthesis frameworks, dental implants, implant abutments, orthodontics brackets, endodontic posts, and surgical instruments.²⁵

Zirconium oxide, also known as zirconia, is a white crystalline oxide of the metal element zirconium. It is processed and purified to produce porous bodies, which can be milled through CAD/CAM with great precision. Zirconia blocks can be milled at three different stages: green, pre-sintered, and fully sintered.²⁶ The original zirconia frameworks milled from green stage and pre-sintered zirconia blocks are enlarged to compensate for prospective material shrinkage (20 percent to 25 percent) that occurs during the final sintering stage.²⁷ The milling of green stage and pre-sintered zirconia blocks are faster and less wear-and-tear producing on hardware than the milling of fully sintered blocks. Due to the increased hardness of the fully sintered zirconia material, they are not subject to dimensional change such as shrinkage after milling. Once densely sintered, a polycrystalline ceramic is produced that does not contain a glass phase like other dental ceramics.

TRANSFORMATION TOUGHENING

Depending on temperature, zirconia crystals can have a monoclinic (M), tetragonal (T), or cubic structure. At high temperature, zirconia has a cubic structure. As temperature is lowered to 2370°C, the atoms rearrange themselves and the structure becomes tetragonal. Then, the tetragonal structure transforms to a monoclinic structure below 1170 °C. The transformation from tetragonal to monoclinic results in a volume

change (4 percent to 5 percent), which makes zirconia stronger and tougher than aluminum oxide. Some oxides such as yttrium oxide (Y_2O_3), magnesium oxide (MgO), calcium oxide (CaO), and others are added to zirconia to stabilize tetragonal crystal structure at room temperature. This partially stabilized zirconia has high flexural strength and fracture toughness.³ A phenomenon of transformation toughening occurs when an increase in the tensile stresses at a crack tip causes the transformation from tetragonal to monoclinic phase, resulting in a localized expansion of 4 percent to 5 percent. Localized expansion triggers compressive stresses at the crack tip, which counteract the external tensile stresses resulting in retarding crack propagation. Thus, the crack is closed until a much higher stress is applied. Yttrium-oxide stabilized tetragonal zirconia polycrystal (Y-TZP) has desirable mechanical properties for restorative dentistry.

ADHESION IN DENTISTRY/RESIN TO ZIRCONIA BONDING

Despite the good mechanical properties of zirconia, another major issue arises pertaining to bonding of ceramic restoration to resin cements.² When bonding ceramic to tooth structure, two interfaces determine the final bond strength of the restoration: dentin-resin cement and ceramic-resin interfaces. Therefore, it is important to ensure optimal bond strength at these interfaces. Wettability of the conditioned adherent surface with resin cement is important for the bonding of ceramics regardless of the mechanism of bonding, for example chemical, micromechanical interlocking, or combination.²⁸ Zirconia is densely sintered and does not contain glass phase; therefore, it cannot be etched with hydrofluoric acid to create micro retentive etching patterns. It does not contain any silica, so silanes cannot be used to promote bonding. Therefore, numerous *in-*

vitro studies have been done on the bonding ability of adhesive systems to zirconia framework material.

A study that compared the shear bond strength of zirconia and dentine using eight different cements has indicated that resin cements produce higher bond strengths than the conventional water-based cements, such as zinc phosphate and glass ionomer, and resin modified glass ionomer cement.²⁹ Another study also confirmed that the bond strength of GIC cement was too low to be applied in the dental practice for the adhesion of zirconia-based restorations after testing for shear bond strength. It was commented that only resin-based luting cements could produce clinically acceptable bond strength even after thermal cycling.²⁸ Another study had evaluated the bonding of zirconia with 11 cements with and without artificial aging and found that the resin cement could result in the formation of durable and strong bonds even after treatment under water storage and thermocycling.³⁰

SURFACE TREATMENTS OF ZIRCONIA

The composition and mechanical properties of zirconia crystalline ceramics differ from those of classic ceramics. So, bonding to zirconia has become a topic of interest. A strong resin bond relies on micromechanical interlocking and chemical bonding to the ceramic surface. To obtain durable retention of zirconia restoration, various surface treatments should be carried out before cementation to improve the bond strength of resin cement to zirconia. Several treatments like sandblasting, acid etching, selective infiltration etching, surface coating, and laser irradiation have been studied in the recent years for adequate surface activation.^{7,12,31}

Acid etching increases the surface area and wettability of silicate-based restorations by changing their surface energy and bonding to resin cements.³² However the microstructure of zirconia ceramic is composed of acid resistant zirconium oxide. Therefore, the acid etching does not produce significant topographic alteration in ceramics with high crystalline content to create durable resin bond strength.^{14,33}

Selective infiltration etching and heat-induced maturation technique was used by Aboushelib et al. to provide strong and durable bonds between zirconia ceramic and composite materials.³⁴ In the selective infiltration etching method, zirconia surface is coated with a thin layer of glass infiltration agent, which consists of silica, alumina, sodium oxide, potassium oxide, and titanium oxide. This glass-conditioning material has similar co-efficient of thermal expansion as zirconia. When the temperature is raised above its glass transition temperature (T_g), the molten glass will diffuse and rearrange zirconia grains and makes a nano-mechanical retentive structure when dissolved in hydrofluoric acid (HF) solution.³⁴

Porcelain coating on zirconia surface followed by acid etching or sandblasting is one of the most frequently used methods. Coating of silica-based ceramics on zirconia ceramics followed by silanization can successfully increase the strength of bonding to composite material.^{35,36} It can be due to the formation of a siloxane network with silica or an increase in the roughness of surface by fusing silica ceramics. However, a study showed reduced tensile bond strength after thermocycling.³⁶ This difference in resin zirconia bond strength can be due to variation in the selection of zirconia and porcelain coating combinations. Apart from porcelain veneering, other methods like adding more silica content on zirconia surface have been brought forward.

It was claimed that the sandblasted (with 50- μm alumina particles) zirconia samples produce higher shear bond strength than others. The treatment of sandblasting was found to result in the loss of surface materials and to increase the surface roughness. However, this technique creates surface micro cracks resulting in apparent decrease in strength, and fracture toughness of the zirconia.³⁷ In 1998 Kern et al. achieved durable bond to airborne particle abraded (110 $\mu\text{ Al}_2\text{O}_3$ at 0.25 MPa) zirconia ceramic after 150 days of water storage with thermocycling using resin composite with a special adhesive monomer. In this study, airborne particle abrasion, silane application and use of Bis-GMA resin cement resulted in an initial bond that failed spontaneously after simulated aging. These findings were verified by a long-term study done by Wegner, in which specimens were subjected to two years of water storage and repeated thermocycling.⁸

As a different surface preparation method for bonding, tribochemical silica coating (Rocatec System) of zirconia ceramics air abraded with Al_2O_3 particles modified with silica has been introduced.^{7,19,30,38,39} The authors indicated that the use of MDP-containing resin cements in conjunction with alumina particles air-abrasion is needed in order to achieve a durable bond. The functional phosphate group of MDP (10-Methacryloxydecyl dihydrogen phosphate) forms a water resistant chemical bond with zirconia. The MDP resin cements are hydrolytically stable and therefore tend not to decrease in bond strength overtime.⁴⁰

It is somewhat debatable that whether ultrasonic cleaning should be carried out after tribochemical silica coating treatment. The ultrasonic cleaning was suggested for enhancing the strength and durable bond between resin cement and titanium⁴¹ but no significant influence was detected when testing the tensile bond strength between resin

and zirconia after 30 days water storage combined with 150 days of thermocycles.⁴² Nishigawa et al. reported a negative effect of ultrasonic cleaning in distilled water on bonding to silica-coated zirconia ceramic as compared to groups that were bonded without ultrasonic cleaning.⁴³ The study demonstrated that the ultrasonic bath in distilled water for 1 min reduced mean shear bond strength. Extending ultrasonic bath time to 5 min even further reduced the shear bond strength. Thus, it was declared that ultrasonic cleaning on tribochemically silanized zirconia should be avoided. The decrease in bond strength was attributed to the fact that ultrasonic cleaning removed loose silica particles, and also a significant amount of silica coating layer from the ceramic surface. However, a negative effect of ultrasonic cleaning in alcohol was not found. Thus, one may speculate that the negative effect on bonding might be related to the effect of water on the highly reactive silica-coated surface rather than to the ultrasonic cleaning itself.

Combined surface treatment with airborne particle abrasion and a specific adhesive monomer with a hydrolytic phosphate monomer have proved for bonding to zirconia ceramics. Thus, several published research articles^{6-8,18,19,31,38,42,44-46} have demonstrated that the combination of surface grinding techniques and traditional resin cementation significantly increases the bond strength of zirconia to resin cement.

CLEANING CONTAMINATED ZIRCONIA

A strong durable resin-ceramic bonding can be achieved through ceramic surface pretreatments in a strictly controlled environment. However, the luting surfaces of ceramic restorations get contaminated with saliva, blood, or silicone indicators during clinical try-in procedures. This contamination significantly affects resin bond strength to zirconia. The ceramic cleaning methods after try-in procedures depends on the type of

contamination.⁴⁷ Attia et al found that cleaning methods after surface conditioning had an insignificant effect on the resin bond to zirconia ceramic after up to 30 days of storage time.⁴²

Silicon contamination is a well-known problem in bonding. A silicon fit indicator is used to check the fit of restoration on tooth and it is believed that a small layer of silicon fit indicator residues are left after intraoral try-in procedures. The main component of silicon disclosing agent is polydimethylsiloxane containing Si-o backbone. During contamination in bonding procedures, organic groups (CH₃) can attach via Si-C bonds to this backbone. A study found that the use of a silicon fit indicator significantly reduced the retention of crowns due to the presence of silicon residual films on the intaglio surface of the crown.⁴⁸ The investigator presumed that chemical reactions and covalent bonds might occur between silicon indicator films and restorations, leading to a stable adherence of silicone to bonding substrate and therefore reducing resin bonding.

Saliva contamination is frequently one of the main reasons for reducing resin bond strength.^{16,37,42,43,47,49-55} Yang et al. found a strong influence of saliva contamination and cleaning methods on resin bonding to zirconia and its durability. In his study, he found that non-covalent adsorption of salivary proteins on roughened “activated” air-borne particle abraded surface occurred during saliva immersion, which could not be removed by water rinsing as showed by XPS. Zirconia has strong affinity to phosphate group, which is found in saliva and other fluids. After saliva contamination, XPS (X-ray photoelectron spectroscopy) analysis revealed an organic coating that resisted complete removal with water rinsing, isopropanol, or with phosphoric acid.⁵⁰

According to Phark and colleagues, conventional contaminants like saliva, blood and die stone play a significant role in bonding to modified zirconia surfaces.¹⁶ They concluded that procedures such as clinical try-ins and laboratory-manufacturing procedures impart a thin layer of contaminants on the surface of the modified ceramic surface that are detrimental to bonding. The mechanism behind the contamination of zirconium oxide surfaces is well explained by Kweon et al.²¹ Zirconium shows a strong affinity towards the phosphate group in that the zirconium surfaces react with phosphoric acid in an acid-base reaction. Consequently, saliva and other body fluids that contain various phosphates groups, such as phospholipids, can react irreversibly with zirconium surface and thus make cleaning a very difficult task.

Zhang found that saliva contamination adversely affects resin bonding to zirconia because it deposits an organic adhesive coating on the restorative materials in the first few seconds of the exposure which is resistant to washing.³⁷ The finding by Aboush study suggested that ceramic surface should be treated with silane before try-in procedures. After intraoral try-in, it is recommend to treat ceramic surfaces with phosphoric acid before applying fresh layers of silane to ensure proper bonding.⁴⁹ But according to Zhang et al, phosphoric acid cleaning effectively removed saliva contamination from coated bonding surfaces, but was not so effective on the removal of the silicone disclosing agent.³⁷ Cleaning with acetone was only effective in the elimination of silicone contaminants, but not for removing salivary residues. Therefore, phosphoric acid or acetone might not serve as an effective cleaning agent. Kern observed a significant decrease in bond strength of resin to zirconia after cleaning with phosphoric acid.

Therefore, factors influencing resin bonding to zirconia ceramic include the wettability of ceramic by adhesive resin, the roughness of ceramic surface, the composition of adhesive resin, the handling performance of adhesive resin, and possible contamination during bonding procedures. Several studies showed different methods to remove contamination but none of the methods have proved to be best. So, the aim of this study was to investigate the effect of saliva contamination and subsequent cleansing methods on zirconia shear bond strength durability with resin cement. Ivoclean, a new cleaning agent, was used to clean saliva contamination, and shear bond strength was determined by a universal testing machine. Failure mode was checked under a light microscope.

MATERIALS AND METHODS

In this *in-vitro* study, the shear bond strength of resin cement to Y-TZP zirconia was evaluated after contamination with saliva and subsequent cleaning methods. The shear bond strength was determined using a MTS Sintech ReNew 1123 universal testing machine (MTS Systems Corporation, St. Paul, MN) 24 h after cementation and after X5000 thermocycles.

SPECIMEN FABRICATION

Eighty square-shaped specimens ($\phi = 12 \text{ mm} \times 12 \times 3 \text{ mm}$) of yttria-stabilized full-contour zirconia (Diazir, Ivoclar-Vivadent, Amherst, NY) were cut from zirconia blocks using a water-cooled saw machine (Isomet 1000, Buehler, Lake Bluff, IL). The obtained specimens were sintered in a high-temperature furnace (Programat® S1, Ivoclar-Vivadent, Amherst, NY)^{56,57} at 1500°C for 8 hours. Then, the specimens were embedded in freshly mixed acrylic resin. (Bosworth Fastray -- Bosworth Co., Durham, England) and allowed to auto polymerize. The bonding surfaces of all specimens were wet-finished with silicon carbide papers (600- to 1200 grit, LECO Corp., Saint Joseph, MI) and cleaned with distilled water.

Surface Modification

All specimens were sandblasted with 50- μm aluminum oxide particles (Patterson Dental Supply, Inc, Saint Paul, MN) for 15 s, under 2.5 bars pressure and from a distance of 10 mm.^{58,59} Then, specimens were rinsed with deionized water for 20 s and air-dried with oil free air for 10 s.

Contamination Protocol and Experimental Design

Following air-abrasion treatment the zirconia specimens were divided into four groups according to the experimental design. Specimens were immersed in 2 mL stimulated saliva (from saliva bank under IRB approval #1303010880) for 1 min with the exception of the control group and divided into four experimental groups according to the cleaning methods, as follows:

Group 1: Control (No saliva contamination)

Zirconia samples not treated with stimulated saliva.

Group 2: Water rinse

After saliva contamination, samples were rinsed under deionized water for 15 s, and then air-dried with oil free air for 10 s

Group 3: Isopropanol

After saliva contamination, samples were immersed in 70 percent isopropanol (Fishers scientific) for 2 min, rinsed with deionized water for 15 sec and then air-dried for 10 s.

Group 4: Ivoclean

After saliva contamination, samples were cleaned with a commercial cleaning paste - Ivoclean (Ivoclar-Vivadent, FL) according to manufacturer's instructions. Shortly, the cleaning paste was applied with a brush and left undisturbed for 20 s. Then, samples were rinsed with deionized water for 15 s and air dried for 10 s.

Bonding Procedure

After the samples received the assigned cleaning regimen a zirconia silane coupling agent (Monobond Plus, Ivoclar Vivadent, Amherst, NY) was applied with a brush on all the samples and left undisturbed for 1 min, and then dried with a stream of air to let the solvent dry.

Two resin cement buttons were built in each sample (n = 160) using a specially fabricated jig for the shear bond strength test (SBS) (Bonding jig, Ultradent, South Jordan, UT). The jig contains a cylindrical mold with 2.38 mm in diameter. A dual-curing resin cement (Multilink – Ivoclar-Vivadent, Amherst, NY) was mixed and applied into the mold according to manufacturer's instructions. (Within working time of 180 s, setting time of 300 s). Bonded specimens were light-cured (LEDemetron II – Kerr Corp., Middleton, MI) for 40 s at 5 mm distance and light intensity of 800 mW/cm² with a hand-held light curing device. The light intensity was monitored with a radiometer (Demetron, Kerr, Orange, CA).

Aging Method

Each main group was divided into two subgroups (n = 10/each). Half of the samples were tested for SBS after 24 h at 37°C (100-percent humidity) and the other half were tested after 5000 thermo cycles (TC, 5°C and 55°C) with dwell time of 30 sec to simulate 1.7 years intraoral conditions.⁶⁰

SHEAR BOND STRENGTH

The shear bond strength was determined using a jig (Ultradent, South Jordan, UT) of universal testing machine (Electropuls E3000 All Electric test instrument, Instron

Industrial Products, Grove City, PA). The load was applied to the adhesive interface until failure at crosshead speed of 1 mm/min. The maximum stress to produce fracture was recorded ($\text{N/mm}^2 = \text{MPa}$) using corresponding software.

FAILURE ANALYSIS

The fractured interfaces on the Y-TZP samples were examined in light microscope at X40 magnification to identify the failure mode as adhesive, cohesive or mixed.

STATISTICAL ANALYSIS

Shear bond strength: Mixed-model ANOVA was used to test the effects of cleaning method (control, isopropanol, ivoclean, and saliva), storage conditions (TC and NC) and their interaction on shear bond strength. Pair-wise comparisons were made using Tukey's method to control the overall significance level at 5 percent.

RESULTS

SHEAR BOND STRENGTH

The mean, standard deviation, and standard error values of shear bond strength (SBS) are summarized in Table III for four different cleaning groups and the two different storage conditions. Cleaning method, storage condition, and their interaction had significant impact on shear bond strength. The overall group comparisons showed that the cleaning treatment with Control (Group 1) and Ivoclean (Group 4) have similar mean SBS (13.43 MPa). While other water rinse (Group 2) (2.56 MPa) and Isopropanol (group 3) (4.02 MPa) did not show comparable results to the control group (Table III).

Cleaning method comparisons: As shown in Table V for the control and Ivoclean cleaning method, (NC) had significantly more shear bond strength than thermocycling (TC). However, the water rinse and isopropanol groups did not show significant difference between two conditions.

Storage condition comparisons: Under storage condition NC, the control method had significantly more shear bond strength than the isopropanol and the water rinse method ($p < .001$). Also, under storage condition NC, the Ivoclean method had significantly more shear bond strength than the isopropanol and the water rinse method ($p < .001$) as shown in TABLE VI.

FAILURE ANALYSIS

GEE method was used to analyze failure mode applied to logistic regression. We classified failure mode into two categories, 'Adhesive' and 'Others (cohesive or mixed)', and compared them. TABLE VII has shown the type of failure modes for all groups with

NC and TC conditions. Failure analysis revealed a large percentage of “Others (mixed or cohesive)” failure modes for the majority of specimens and a small percentage of “adhesive” failure at the ceramic resin cement interface. Since the number of event ‘adhesive’ is very small (Table VII), the effects of cleaning methods and storage conditions were addressed, and the interaction effects could not be tested. Overall, cleaning methods had a significant impact on the adhesive mode.

STATISTICAL ANALYSIS

Shear bond strength: Mixed-model ANOVA was used to test the effects of the cleaning method (control, saliva, isopropanol, Ivoclean), storage condition (thermocycling-TC and non-thermocycling-NC) and their interaction on shear bond strength (Table IV). The test result revealed that the cleaning method ($p < .0001$) and storage conditions ($p < .0001$) had a significant influence on the bond strength. While interaction between the group and the conditions was not significant ($p = 0.0001$), indicating that the condition comparisons are valid for all groups and that the group comparisons are valid for all conditions. Pair-wise comparisons were made using Tukey’s method to control the overall significance level at 5 percent.

TABLES AND FIGURES

TABLE I

Materials used and their characteristics

Materials		Manufacturer	Composition
Zirconia	Diazir Full-Contour	Ivoclar- Vivadent, Amherst, NY, USA	Y-TZP
Cleaning agent	Isopropanol	Fishers Scientific	70% Isopropanol
Cleaning Paste	Ivoclean	Ivoclar- Vivadent, Amherst, NY, USA	Zirconium oxide, water, polyethylene glycol, sodium hydroxide, pigments, additives
Silane	Monobond Plus	Ivoclar- Vivadent, Amherst, NY, USA	Alcohol solution of silane methacrylate, phosphoric acid, methacrylate and sulfide methacrylate
Resin Cement	Multilink Automix	Ivoclar- Vivadent, Amherst, NY, USA	Monomer matrix consist of - Dimethacrylate, HEMA, In organic fillers - barium glass, ytterbium trifluoride, spheroid mixed oxide

TABLE II

Zirconia specimen groups and study design (n = 80)

Group 1 (N = 20)		Group 2 (N = 20)		Group 3 (N = 20)		Group 4 (N = 20)	
Control - No saliva contamination		Saliva contamination		Saliva + 70% Isopropanol for 2 min		Saliva + Ivoclean® for 20 sec	
Storage conditions		Storage conditions		Storage conditions		Storage conditions	
24 h (N = 10)	TC (N=10)	24 h (N=10)	TC (N=10)	24 h (N=10)	TC (N=10)	24 h (N=10)	TC (N=10)
N = 20	N = 20	N = 20	N = 20	N = 20	N = 20	N = 20	N = 20

TABLE III

Evaluate the effect of cleaning method and storage condition on shear bond strength – summary table

Cleaning Method	Storage Condition	N	Mean (S.D)
Control	NC	20	11.42 ± 4.30
	TC	20	3.66 ± 2.41
Isopropanol	NC	20	4.02 ± 3.47
	TC	20	3.50 ± 1.83
Ivoclean	NC	20	13.43 ± 10.15
	TC	20	6.48 ± 5.94
Saliva	NC	20	2.56 ± 1.37
	TC	20	3.93 ± 2.88

TABLE IV

Evaluate the effect of cleaning method and storage condition on shear bond strength - ANOVA table

Effect	Num DF	Den DF	F Value	P Value
Cleaning	3	80	15.27	<.0001
Storage	1	80	18.00	<.0001
Cleaning*Storage	3	80	7.82	0.0001

TABLE V

Evaluate the effect of cleaning method and storage condition on shear bond strength – cleaning method comparison

Comparison	Difference	Standard Error	P value
Control: NC > TC	7.76	1.63	0.0002
Isopropanol: NC & TC n.s.	0.52	1.63	1.0000
Ivoclean: NC > TC	6.95	1.63	0.0014
Saliva: NC & TC n.s.	-1.37	1.63	0.9904

TABLE VI

Evaluate the effect of cleaning method and storage condition on shear bond strength – storage condition comparison

Comparison	Difference	Standard Error	P-value
NC: Control & Ivoclean n.s.	-2.01	1.63	0.9206
NC: Control > Isopropanol	7.41	1.63	0.0005
NC: Control > Saliva	8.86	1.63	<.0001
NC: Isopropanol & Saliva n.s.	1.45	1.63	0.9863
NC: Isopropanol < Ivoclean	-9.42	1.63	<.0001
NC: Ivoclean > Saliva	10.87	1.63	<.0001
TC: Control & Isopropanol n.s.	0.16	1.63	1.0000
TC: Control & Ivoclean n.s.	-2.82	1.63	0.6690
TC: Control & Saliva n.s.	-0.27	1.63	1.0000
TC: Isopropanol & Ivoclean n.s.	-2.99	1.63	0.6032
TC: Isopropanol & Saliva n.s.	-0.44	1.63	1.0000
TC: Ivoclean & Saliva n.s.	2.55	1.63	0.7716

TABLE VII

Mixed model for comparison of failure mode adjusting for cleaning method and storage condition – summary table

Cleaning Method	Storage Condition	Failure Mode	N (%)
Control	NC	Others	19 (95.00)
		Adhesive	1 (5.00)
Control	TC	Others	15 (83.33)
		Adhesive	3 (16.67)
Isopropanol	NC	Others	13 (65.00)
		Adhesive	7 (35.00)
Isopropanol	TC	Others	15 (75.00)
		Adhesive	5 (25.00)
Ivoclean	NC	Others	18 (94.74)
		Adhesive	1 (5.26)
Ivoclean	TC	Others	15 (100.00)
		Adhesive	0 (0.00)
Saliva	NC	Others	12 (70.59)
		Adhesive	5 (29.41)
Saliva	TC	Others	14 (77.78)
		Adhesive	4 (22.22)

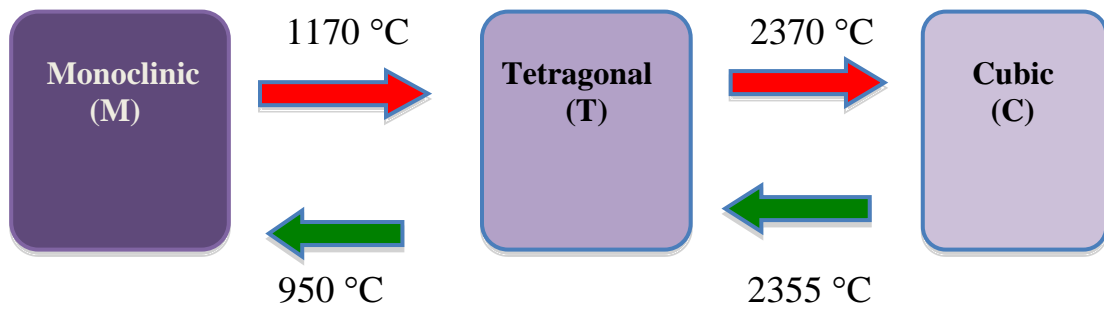


FIGURE 1. Transformation toughening of Zirconia.



FIGURE 2. Polished Zirconia samples.



FIGURE 3. Ivoclean.

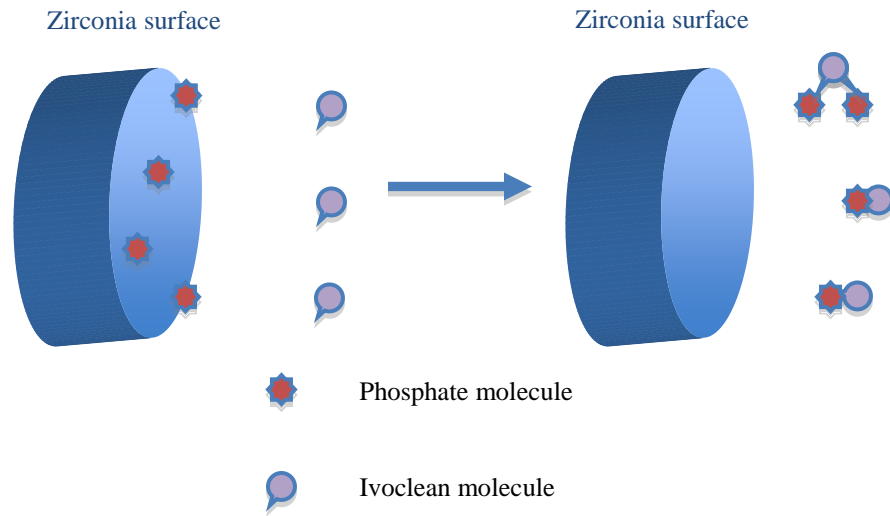


FIGURE 4. Ivoclean – Mechanism of action.



FIGURE 5. Monobond plus, Ivoclar-Vivadent, Amherst, NY.



FIGURE 6. Multilink Automix, Ivoclar-Vivadent, Amherst, NY.

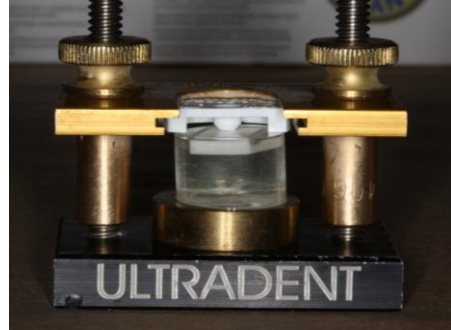


FIGURE 7. Bonding of Zirconia sample with Ultradent Jig.



FIGURE 8. Illustration of bonded Zirconia sample.

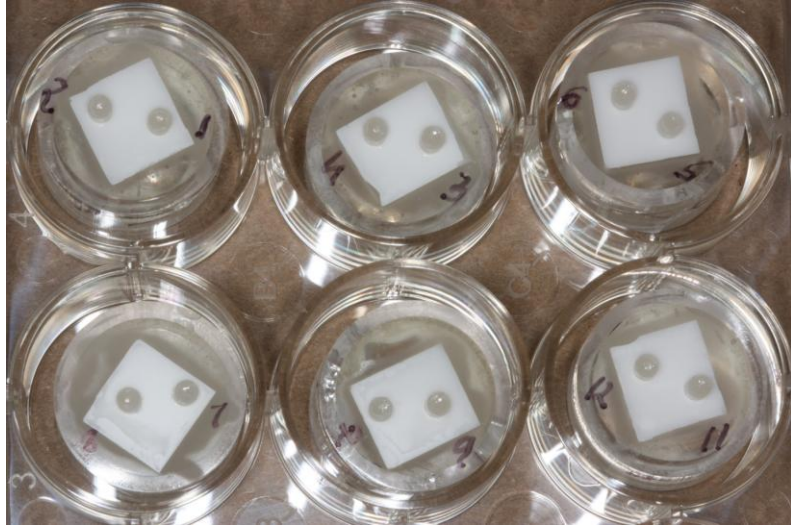


FIGURE 9. Illustration of storage of bonded specimen for each group.



FIGURE 10. Thermocycling machine.

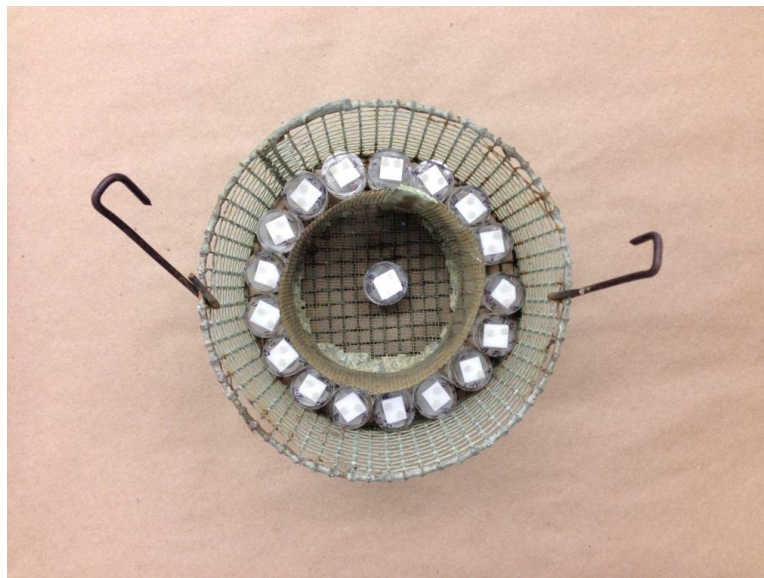


FIGURE 11. Illustration of samples arranged in thermocycling basket.

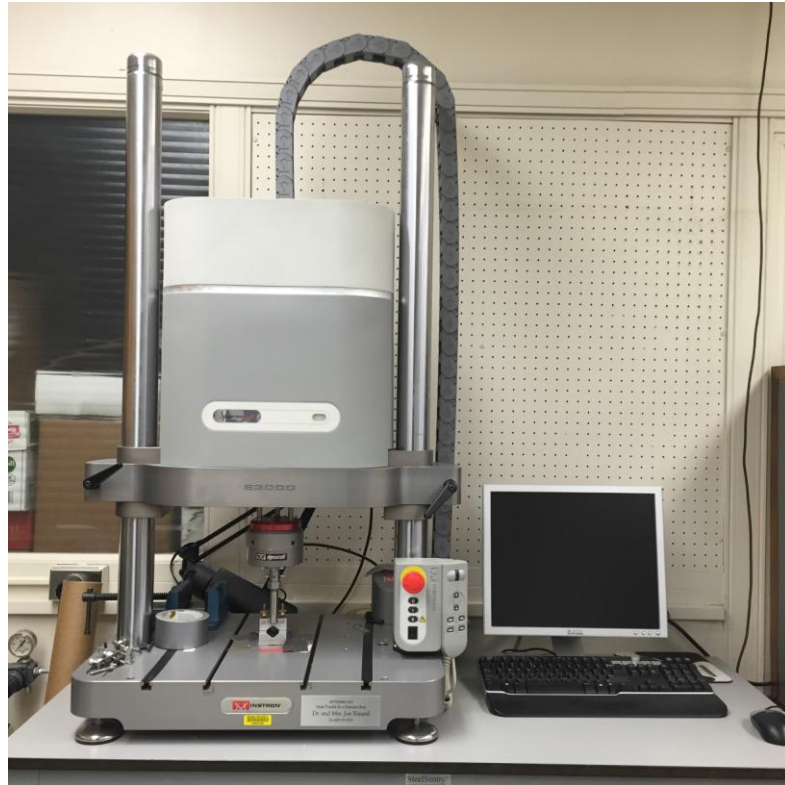


FIGURE 12. Electropuls E3000, All electric test instrument, Instron Industrial Products, Grove City, PA.



FIGURE 13. Illustration of samples arranged in Ultradent jig for shear bond strength testing.



FIGURE 14. Illustration of sample going for shear bond strength test in universal testing machine

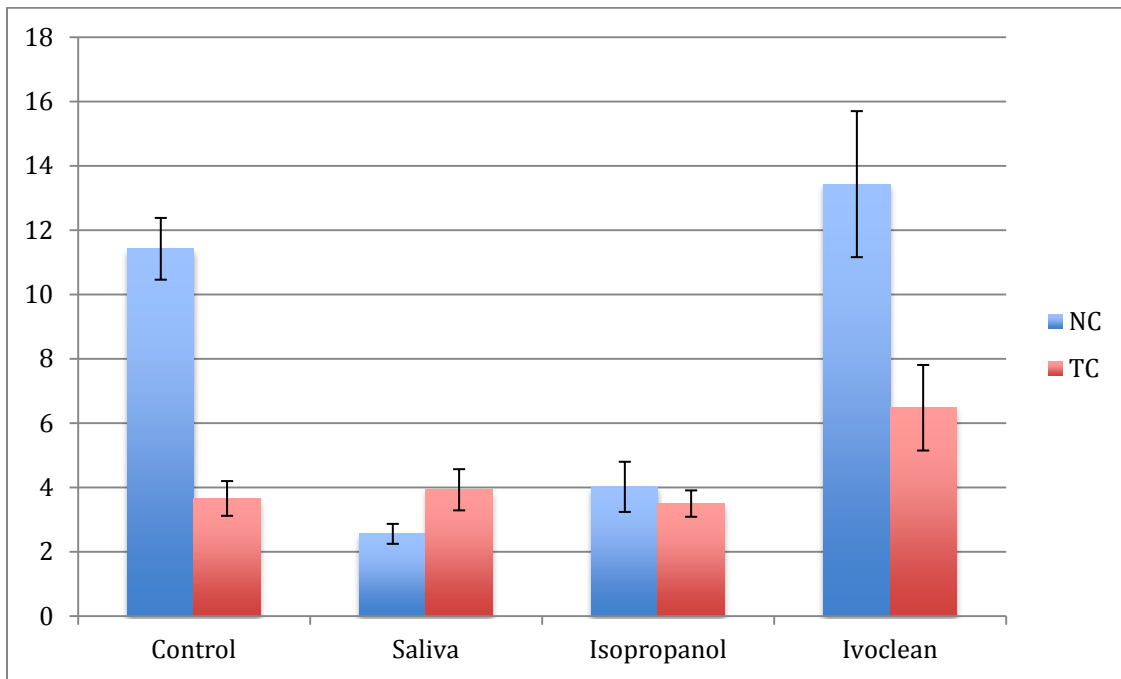


FIGURE 15. Shear bond strength means and standard error for all the groups in NC and TC Condition.

DISCUSSION

In prosthodontics a strong adhesion provides high retention, improves marginal adaptation, prevents the micro infiltration, and increases the fracture strength of the restored tooth and its restoration. This kind of bonding is based on micromechanical interconnection and chemical adhesion of the adhesive to the ceramic surface, which requires the creation of roughness and adequate cleaning to ensure surface activation.

The challenge in promoting a strong and reliable bond between the internal surface of zirconia restoration to the resin luting agents lies on achieving bonding surface free of contaminants that often results from intraoral try-in procedures. The present study evaluated the effect of different zirconia surface cleaning methods (water rinse, isopropanol and a cleaning paste) after saliva contamination on the bond strength to resin cement. The study was performed under 24 hrs of water storage and X5000 thermocycles to simulate intraoral condition.

Previous studies have reported different cleansing protocols, such as water,⁵⁰ alcohol (70%-96% Isopropanol),^{37,47,50,54} phosphoric acid,^{16,37,43,47,50} and additional airborne particle abrasion (Al_2O_3).^{50,52,61} Storage and debonding conditions have been used in previous studies as well.^{19,47,52,54}

In the present study, the values of bond strength of resin cement to zirconia significantly decreased after saliva contamination (2.56 ± 1.37 MPa) compared with the controls (11.42 ± 4.30 MPa). The result of the present study showed a significant effect of aging protocol. The group comparison after 24 h showed that all groups presented lower results after TC. The predominant failure mode of the saliva group was adhesive

mode after TC, which shows that surface contamination of zirconia ceramic with saliva is related to the decreased bond strength. This result is in the agreement with the study done by Quaas et al. The study was designed to test the resin-ceramic bond strength and its durability related to the cleaning methods of contaminated ceramic bonding surface. They found that no cleaning after the contamination group led to the lowest bond strength values.^{51,52}

Saliva contaminations adversely affect the resin bonding because organic deposits remain on the restorative material after few seconds of exposure in saliva.⁶² The prior studies^{43,50} reported that water rinsing may not be effective to remove some saliva contaminants from zirconia surface. Saliva contains 99 percent water combined with small amount of proteins, glycoprotein, sugar, amylase and inorganic particles. Non-covalent adsorption of salivary proteins occurs on the restorative surface after saliva contamination, creating a thin residual film of organic protein that can not be removed with water. This results in decreased bond strength and the inability to establish the bond strength of uncontaminated zirconia. It prevents chemical bonding to zirconia ceramics, while thermocycling then further interferes with the formation of a durable bond. Lower bond strength values and a high percentage of adhesive failure modes can be explained by the fracture phenomena at the surface area of zirconia ceramics.

Initially, 37-percent phosphoric acid was used as one of the cleaning methods. However, in the present study the samples were debonded before the mechanical test (SBS). It can be due to the remaining phosphorous residues, which change the surface-free energy of the ceramic surface for bonding results in negatively impairs bonding ability.

Cleaning with isopropanol was not effective in removing saliva contamination as shown by the fact that both initial bond strength (4.02 ± 3.47 MPa) and bond strength after TC (3.50 MPa) were remarkably lower than the control group. The fracture mode after the bond strength test was adhesive failure, indicating the durability of resin bonding to zirconia ceramic was not satisfactory. Cleaning with isopropanol was only effective in the elimination of silicone contaminations, but not for removing salivary residues.

Some authors^{50, 61} suggested that an additional particle abrasion may provide better bonding results after saliva contamination as compared with the group without contamination. However, mechanical treatments of zirconia, such as sandblasting or grinding, should be done cautiously, because these can negatively influence mechanical properties by inducing compressive stresses or phase transformation on the surface, which increase the strength but at the same time induces flaws and other defects.

Saliva consists of phosphate groups in the form of phospholipids, which actively bond to the intaglio surface of the zirconia restoration. Recently, a new cleaning agent called Ivoclean has come to the market. Ivoclean is an alkaline suspension of zirconium oxide particles. According to the manufacturer's scientific documentation, Ivoclean contains zirconia, water, polyethylene glycol, sodium hydroxide and other additives. Due to the size and concentration of particles in the medium, phosphate contaminants from saliva are more likely to bond to the particles in the Ivoclean than ceramic surfaces, leaving behind a clean zirconium oxide surface. In the present study, the Ivoclean group showed bond strength results (13.43 MPa), comparable to the control group (11.42 MPa) in non-thermocycling conditions. Even though thermocycling reduced the values of shear

bond strength, the results showed that the Ivoclean group maintained values comparable to the those of the control group.

Therefore, the null hypothesis that cleaning methods or storage conditions will not influence bonding to zirconia should be rejected. The cleaning methods employed after saliva contamination positively influenced resin bonding to zirconia. More specifically, the shear bond strength of resin cement to zirconia was improved after cleaning saliva-contaminated samples with Ivoclean both immediately and after thermal aging (thermocycling).

SUMMARY AND CONCLUSION

Within the limitation of this *in vitro* study the following conclusions can be made:

1. Zirconia ceramics' cleaning protocol must be considered after exposure to saliva during intraoral try-in procedures.
2. The new cleaning paste Ivoclean applied on the contaminated zirconia surface is the most effective method, comparable to the control group.

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ABSTRACT

INFLUENCE OF SALIVA CONTAMINATION ON RESIN BOND DURABILITY
TO ZIRCONIA – EFFECT OF CLEANING METHODS

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Background and Rationale: As compared with glass-based ceramics, zirconia has gained considerable popularity in restorative dentistry due to its superior mechanical properties.^{2,31,42} Clinically, however, zirconia ceramics pose a significant challenge regarding the achievement of a reliable and durable bond to resin-based cements. Thus far, it has been established that zirconia bond to resin-based cements can be enhanced after different surface conditioning methods, such as airborne particle abrasion with

aluminum oxide particles. Meanwhile, another major issue pertaining to bonding of ceramic restorations is related to its potential contamination before cementation. Briefly, after sandblasting and clinical try-in procedures, zirconia can be contaminated with saliva and/or blood. As with many metals, zirconium shows a strong affinity towards the phosphate group found in saliva and other fluids, which reacts with the zirconia surface and makes bonding very difficult. Recently, a new cleaning agent called Ivoclean[®] (Ivoclar-Vivadent), which is an alkaline suspension of zirconium oxide particles, has been introduced in the market to remove contamination from zirconia in an effort to improve bonding to resin cements.

Objective: The purpose of this study was to evaluate the influence of saliva contamination and the effect of several cleaning methods, including Ivoclean on resin bond strength to zirconia. Materials and Methods: Eighty square-shaped specimens ($\phi = 12 \text{ mm} \times 12 \text{ mm} \times 3 \text{ mm}$) of yttria-stabilized full-contour zirconia (Diazir[®], Ivoclar-Vivadent, Amherst, NY) were sectioned from zirconia blocks using a water-cooled diamond blade. Then, these specimens were embedded in acrylic resin, and their surfaces gradually finished with silicon carbide papers (600 grit to 1200 grit). The prepared zirconia surfaces were sandblasted with 50- μm aluminum oxide particles for 15 s, under 2.5 bars and from distance of 10 mm. After sandblasting the specimens were cleaned in an ultrasonic bath containing distilled water for 5 min and air-dried for 10s. All samples were equally divided into 4 groups ($n = 20$) according to the cleaning method. Airborne particle abraded specimens without contamination was served as the control group. Remaining groups were contaminated with saliva, and subjected to different cleaning protocols, namely: Ivoclean[®], 70% isopropanol, and no treatment. Two resin cement

buttons (Multilink – Ivoclar-Vivadent, Amherst, NY) were built over each zirconia surface and light-cured following the manufacturer recommendations. The influence of contamination and surface cleaning methods on ceramic bond durability were examined after 24 h on half of the samples in each group (n = 10, n = 20), and the other half (n = 10, n = 20) specimens will undergo 6000 thermocycles (TC) before shear bond testing in the universal testing machine. Conclusion of Expected Outcomes: The shear bond strength of resin cement to zirconia led to a significant improvement after cleaning with Ivoclean both immediately and after thermal aging.

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Professional Organizations

ACP – The American College of Prosthodontists
JFJ – The John F. Johnston Society
CDS – Chicago Dental Society
IDA – Indian Dental Association
ADA – American Dental Association