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The Effect of Different Surface Treatments on The Bond Strength of Zirconia

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Abstract

Objectives: The purpose of the study was to evaluate the influence of various surface treatment methods on the resin bond durability of zirconia.

Methods: Hundred KATANA Zirconia STML (n=20) specimens were sectioned and sintered in an induction furnace (CEREC SpeedFire, Dentsply Sirona, Germany). Specimen surfaces were ground finished with 800 grit silicon carbide abrasive with cooling water and cleaned in an alcohol bath ultrasonically before cementation for 5 min, and thoroughly washed again with running distilled water. Specimens in Group 1 had no surface treatment, Group 2 was air-abraded with 50 µm aluminum oxide, Group 3 was air abraded with glass bead particles, Group 4 specimens were immersed in Zircos E etching solution for 2 h and Group 5 specimens were immersed in 48% Hydrofluoric acid solution heated at 25oC for 30 min. Cylindrical composite resin specimens (2.1 mm in diameter, 3 mm in height) were bonded to the zirconia samples with self-adhesive resin cement Panavia V5 (PV5, after application of ceramic primer) following manufacturers' instructions. A load of 1000 g was applied to the composite cylinders during bonding in an alignment apparatus, then light cured for 80 s.

Each main group was divided into two subgroups (n = 10/each). Half of the samples were tested for SBS after 48 h in distilled water at 37o C (100-percent humidity), 10

then subjected to 10,000 thermo cycles and the other half were tested after 10,000 thermo cycles.

Shear bond strength was determined using a universal testing machine at a crosshead speed of 0.5 mm/m expressed in MPa. The fractured surfaces of specimens were inspected with a stereo microscope and classified as adhesive, cohesive, or mixed failures. One-way ANOVA test and paired t- test was applied for statistical analysis.

Results: All four surface treatment methods tested were significantly different from each other and with the control group (no surface treatment). Mean shear bond strength values for group1 8.316(SD 1.953), group 2 14.976(SD 3.189), group 3 9.286(SD 0.985), group 4 4.831(SD 0.468), and group 5 14.796(SD 0.829). The mean shear bond strength values for all the groups decreased significantly after thermocycling.

Conclusions: According to the results of this in vitro study, air abrasion and heated hydrofluoric acid proved to be better methods for surface treatment of zirconia as compared to other methods. Newly launched zirconia etching solution containing hydrofluoric acid (HF), hydrochloric acid (HCl), sulfuric acid (H2SO4), nitric acid (HNO3), and phosphoric acid (H3PO4) performed poorly as surface treatment agents in increasing the bond strength of zirconia.

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Markus B Blatz, DMD, PhD

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The Effect of Different Surface Treatments on The Bond Strength of Zirconia

Thesis

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By

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Advanced Education Program in Prosthodontics

Department of Preventive and Restorative Sciences

University of Pennsylvania, School of Dental Medicine

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Dedication

First of all, I would like to thank Waheguru ji and Sai baba ji for his blessings and for all the achievements in my life.

I want to dedicate this manuscript to my father, **Mr. Harinder Singh and Mrs. Neelam**, my parents, to whom this thesis is dedicated with all my love, respect, and gratitude. Without my parents' boundless love and care, neither my life story could have been written nor could this thesis. I would like to deeply thank my sisters, **Dr. Ramandeep and Ripandeep,** for their love and support throughout my studies and my brother in laws, **Dr. Jayant Mudgal and Jaldeep Rekhi** for their encouragement. A big thanks to my In-laws, **Dr. Rakesh Chawdhry** and **Dr. Meena Chawdhry** for believing in me.

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Literature review and Introduction:

The substantial increase in esthetic consciousness and patient demand for esthetic dental restoration has led to a rapid development in the art and science of restorative dental materials. Superior esthetic requirements are no longer a luxury. It is the everyday basic need that has pushed dental materials to the edge of its limitations. Ceramics are material of choice for such restorations because they closely mimic the optical properties of enamel and dentine, in addition to their chemical and mechanical properties such as biocompatibility, high elastic modulus, low thermal expansion coefficients, and good wear resistance[1].

Increased application of ceramic restorations has led to development of a variety of ceramic systems. Demand for improved clinical performance pressured the dental material industry to introduce several ceramic materials that are classified by porcelain type as feldspathic porcelain, leucite reinforced, aluminous, glass-infiltrated, glass infiltrated spinell, glass-infiltrated zirconia and glass-ceramic. Interest for using high-strength zirconium oxide ceramics for the fabrication of computer-manufactured full coverage crowns and bridge frameworks is growing in recent years, due to their improved mechanical properties in comparison to more conventional alumina or lithium disilicate-based ceramics. [2-4]

Zirconia

Zirconia has a monoclinic crystal structure at room temperature and a tetragonal and cubic structure at increasing temperatures. Formulations used in dentistry contain mainly tetragonal crystals that are partially stabilized with yttrium oxide (Y_2O_3) and have a flexural strength of 900 to 1,400 MPa, a modulus of elasticity of 210 GPa, and a fracture toughness of 10 MPa/m. Pure zirconia exhibits a polymorphic phase transformation from a cubic to a tetragonal to a monoclinic phase accompanied by a high-volume change when cooling down from high temperatures after sintering which makes the sintered body unstable. However, the addition of 3–6 wt% Y_2O_3 stabilizes the zirconia in the tetragonal phase which is otherwise not stable at room temperature. Zirconia with Y_2O_3 added in concentrations less than those required to stabilize it completely is called yttrium-oxide–partially-stabilized zirconia (YPSZ) when the sintered ceramic contains a mixture of cubic and tetragonal phases. YPSZ has optimized physical properties and exhibits much more fracture toughness and fracture strength than alumina ceramic.[5,6]

Properties termed active crack resistance or transformation toughening[7] are unique to this material: external stresses and cracks cause transformation of the tetragonal particle into a monoclinic one with greater volume (approximately 3% to 5%), subjecting a crack under compressive stresses and impeding its growth. However, the actual effects of this phase transformation on ultimate strength and its role in an accelerated aging process (low- temperature surface degradation) are discussed controversially.

Since the late eighties, YPSZ has been used in orthopedics for total hip replacement. In dentistry, YPSZ has been successfully tested in animals as a material for oral implants. Recently, YPSZ posts for restoring endodontically treated teeth have been introduced and YPSZ abutments for dental implants are currently being developed.[5] The use of zirconia in restorative dentistry has grown exponentially over the past decade. Early zirconia formulations were used for frameworks because of their high flexural strength and unesthetic opacity in porcelain-fused-to-zirconia restorations. Through processing refinements, materials with increased translucency were introduced so that veneering with feldspathic porcelain was not required and the material could be used in monolithic form.[5,6,8]

Restorations are typically milled from green-stage or pre-sintered (white-stage) zirconia blocks before full sintering. Only very few CAD/CAM systems mill from fully sintered blocks, which have a significantly higher hardness and flexural strength, making the milling process time-consuming and taxing on the milling equipment. First-generation conventional zirconia copings and frameworks are veneered with feldspathic ceramic (porcelain-fused-to-zirconia, PFZ) for esthetic reasons since they are rather opaque and monochromatic white. Early studies indicate a high incidence of veneer fractures and chippings.[9,10] The development of veneering ceramics that better matched the thermal (coefficient of thermal expansion [CTE]) and physical properties of zirconia as well as firing and cooling protocols to control internal thermal stresses significantly increased reliability of PFZ restorations.[11] More recent investigations show long-term success rates of PFZ crowns that are not different from metal- ceramics.[11,12] Nevertheless, concerns about possible veneering ceramic fractures made monolithic full-contour restorations the predominant all-ceramic choice. A fully digital CAD/CAM process has made full-contour zirconia (FCZ) restoration fabrication highly predictable and cost-effective. Second-generation zirconia materials have a higher translucency and slightly lower flexural strength than conventional zirconia. A customized, tooth-like appearance is created through infiltration of liquid dyes in a green or pre-sintered stage and firing of stains and glazes after sintering. Some manufacturers offer pre-shaded and even multilayer zirconia blanks that mimic natural tooth appearance and can be further customized.[6]

The latest generation of zirconia features significantly greater light transmission with optical properties suitable even for anterior teeth. The higher translucency is achieved by slight changes of the Y_2O_3 content (5 mol-% or more instead of 3 mol-%), resulting in a higher amount of cubic-phase particles.[13,14] However, the flexural strength (between 550 and 800 MPa) is significantly lower than that of conventional zirconia but still considerably higher than any silica-based ceramic. Some clinicians have begun using FCZ for resin-bonded partial-coverage inlays/onlays and laminate veneers.[15]

Bonding of Zirconia

In the literature, historically, there was limited data regarding bonding methods to zirconia ceramic. Hydrofluoric etching and silanization, which enhances the resin bond to conventional silica-based ceramics, does not improve the resin bond strength to alumina or zirconia ceramics. However, it has been shown that silica coating followed by silanization can be successfully used for bonding glass-infiltrated alumina ceramic. In the same study, phosphate monomer-containing resin composites were successfully bonded to sandblasted alumina ceramic, while using a conventional Bis-GMA resin composite a durable resin bond to alumina ceramic was not achieved.

With emerging long-term studies, evidence now suggests that air particle abrasion followed by application of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) containing primer and composite resin cement help achieve a robust bond between zirconia and tooth structure.[16,17]

Composite resin cements and compatible bonding systems increase retention, improve marginal seal and fracture strength of all-ceramic restorations. While considered “cementable,” some zirconia restorations benefit from insertion with composite resin-cement systems. These include zirconia restorations that are less strong, are thin, lack retention, or rely on resin bonding, such as resin-bonded fixed prostheses, high translucency or ultra-high translucency zirconia or bonded laminate veneers.[16,18] The success of resin bonding relies on the proper materials selection and adequate treatment of tooth and restoration bonding surfaces.[19,20]

Resin cements and resin ceramic bonding

Adhesive bonding techniques and modern all-ceramic systems offer a wide range of highly esthetic treatment options.[21-25] Bonding to traditional silica- based ceramics is a predictable procedure yielding durable results when certain guidelines are followed.[11,26] However, the composition and physical properties of high-strength ceramic materials, such as aluminum oxide-based (Al_2O_3)[27-31] and zirconium oxide-based (ZrO_2) ceramics,[32] differ substantially from silica-based ceramics [28,33,34] and require alternative bonding techniques to achieve a strong, long-term, durable resin bond.

Resin-based composites are the material of choice for the adhesive luting of ceramic restorations[35]. Composite cements have compositions and characteristics similar to conventional restorative composites and consist of inorganic fillers embedded in an organic matrix (for example: Bis-GMA, TEGDMA, UDMA). Composite cements can be classified according to their initiation mode as auto-polymerizing (chemically activated), photoactivated, or dual-activated materials.

Photoactivated composites offer wide varieties of shades, consistencies, and compositions[35]. Clinical application is simplified through long handling times before and rapid hardening after exposure to light. Shade, thickness, and transmission coefficient of the bonded ceramic restoration and the composite itself influence the conversion rate of the photo-activated material and limit its application to thin silica-based ceramics.

Dual-activated composites offer extended working times and controlled polymerization,[35] although chemical activators ensure a high degree of polymerization. Most dual-activated resin cements still require photopolymerization and demonstrated inferior hard- ness when light polymerization was omitted.[36,37] Various dual-activated resin cements showed no differences in resin-bond strengths between glass ceramics and enamel.[38] Auto-polymerizing resin cements have fixed setting times and are generally indicated for resin bonding metal-based or opaque, high-strength ceramic restorations[35].

Self-adhesive resin cements were developed based on the chemistry of resin cements and self-etch adhesives. They etch, prime, and bond to dentin without the need of separate agents for each of these steps. Therefore, the application is very simple, and the concept of the smear layer as a bonding substrate has been reintroduced with the expectation of a low incidence of postoperative sensitivity and pulp response.[39-42]

These cements provide good bond strengths with less steps making them more user friendly.[42] Their application can be accomplished in a single clinical step, similar to cementation procedures with conventional luting agents. Besides simplified application techniques, they seem less susceptible to moisture contamination. A low incidence of postoperative sensitivity is expected with these luting agents.[43]

Due to these properties and widespread applications, these cements have become popular in clinical use.

However, consensus and one of the latest reviews of literature on adhesive systems used in Indirect restorations cementation suggest that the 3-step system was the most effective due to its lower risk of hydrolytic degradation at the interface level. Unfortunately, it is a highly sensitive technique, which is why more humidity control is suggested depending on the detailed components by the manufacturer.

The self-etched adhesive systems reduce the time spent in clinical practice. However, at the interface they behave as permeable membranes, which facilitates the passage of fluids from oral environment to dentin and vice versa (dentine–intraoral environment), being more susceptible to degradation. Furthermore, its use is limited when using dual and self-curing cements, as its components can interfere with the polymerization process. They are also prone to form a discontinuous, irregular, and shallow hybrid layer associated with low wettability, viscosity of the system, and low infiltration into the dental tissues.[44]

Thus, the 3-step adhesive systems still are the gold standard for the cementing of indirect restorations.

Surface modification methods

It has been reported that the clinical success of resin bonding procedures for cementing ceramic restorations and repairing fractured ceramic restorations depends on the quality and durability of the bond. The former depends upon the bonding mechanisms that are controlled in part by the surface treatment that promotes micromechanical and/or chemical bond to the substrate. The nonreactive surface of zirconia (acid-resistant ceramic), however, presents a consistent issue of poor adhesion, i.e., low bond strength to other substrates.[45-50]

As zirconia is considered as an acid-resistance ceramic, other methods to produce micromechanical retention have been used, including airborne particle abrasion (APA) systems, often called sandblasting with alumina or silica-modified alumina particles, glass beads, abrasion with diamond rotary instrument, laser abrasion, acid etching, or a combination of these techniques. Several studies[51-58] reported that airborne particle abrasion methods using alumina particles or silica-modified alumina particles (silica coating) produced greater surface roughness (Ra) values and that silica coated surfaces showed a significant increase (76%) in the concentration of silicon, which should enhance bonding to resin via silane coupling agents.[52,55,57] Therefore, silica coating (silicatization) systems (e.g., Rocatec and Cojet, 3M-ESPE) have been used in the past to create a silica layer on metal and ceramic surfaces through high-speed surface impact of the silica-modified alumina particles that can penetrate up to 15µm into ceramic and metal substrates. This tribochemical effect may be explained by two bonding mechanisms: (1) the creation of a topographic pattern via airborne particle abrasion allowing for micromechanical bonding to resin; and (2) the promotion of a chemical bond between the silica coated ceramic surface and the resin-based material, via a silane coupling agent.[52,59]

Therefore, the adhesion between dental ceramics and resin-based composites is the result of a physio-chemical interaction across the interface between the resin (adhesive) and the ceramic (substrate). The physical contribution to the adhesion process is dependent on the surface treatment and topography of the substrate and can be characterized by its surface energy. Alteration of the surface topography results in changes on the surface area and on the wettability of the substrate, which are related to the surface energy and the adhesive potential.[45,47,48,60] 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.[45,47-50,60,61]

Though all these methods have been considered for increasing the bond strength of zirconia still every method has flaws. For instance, silica coatings are reportedly insufficient for long-term stability due to the hydrolytic degradation of silica coatings.[62,63] Selective infiltration etching has a couple of clinical problems, including its complexity and the high costs that are associated with the application process. Laser etching is also reportedly less efficient at altering the surface of zirconia than is AB, exhibits lower adhesive strength when dental resin cements are applied, and causes phase transformation into the excessive monoclinic phase.[64,65] It is expected that if AB and 10-methacryloyloxydecyl dihydrogen phosphate- (10-MDP-) containing luting agents are used adequately for cementing zirconia, then this will yield successful long-term clinical bonding.[6,17,66-68] However, it has also been suggested that the surface roughness of zirconia varies according to the particle size, distance, and duration of AB, which are manual processes and may affect the bonding strength of the resin adhesive.[69] In addition, a few studies have reported a decrease in the physical strength of zirconia depending on the flaws caused by AB.[70-72]

Though it has been proposed that 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, however, a study by Sriamporn revealed micro-morphologic changes on the surface topography of zirconia after applying 9.5%

hydrofluoric acid (HF) in 25°C for 24 h, 9.5% HF in 80°C, and 48% HF in 25°C and concluded that concentration and temperature of acid could affect the reaction rate.[73] Zhang Q et al (2020)[74] also compared shear bond strength of zirconia samples with surface treatment by HF and sandblasting. They concluded that shear bond strength of samples etched with HF was comparable to the samples that were sandblasted. HF etching creates a nano roughness surface that significantly increases SBS and surface hydrophilicity, with the minimal damage to zirconia. Thus, the role of different concentration of strong acids to change the surface topography of zirconia to increase its bond strength cannot be ruled out. Recently, Zircos E etching system (ZSAT: Zirconia Surface Architecturing Technique, M&C Dental Co., Eunjin Chemical Co., Seoul, Korea), a mixture of nitric acid and hydrofluoric acid that could be applied in room temperature, has been introduced which claims to change the surface topography of zirconia which is conducive to increase its bond strength. [75]

Various methods of surface treatment of zirconia have been proposed in the literature to increase the bond strength, still the best method remains ambiguous.

Measuring bond strengths

The shear strength is the maximum stress that a material can withstand before failure in a shear mode of loading.

Bond strength tests are abundant in the dental literature in part because they are relatively easy to perform and are not equipment intensive. They, however, present several limitations that reduce their usefulness as a selection criterion in clinical practice. These drawbacks are briefly explained below.

Macro Shear Bond Strength Tests

In a macro shear bond strength test,[76] a composite cylinder is built on the bonding substrate. After a pre- determined storage time, the specimen is positioned in a universal testing machine where a single-edged chisel, a flat-end rod, or a wire loop is attached to the actuator used to dislodge the composite cylinder from the substrate.

It is important to note that in shear tests, it is a tensile stress that actually causes debonding. In other words, the term shear test refers to the loading mode, and not to the stress causing interfacial failure. When the loading distance from the interface increases, tensile stress also increases due to creation of a bending moment in the composite cylinder.

The location and configuration of the loading device influences the stress distribution at the bonded interface and therefore affects the bond strength. Computer simulation using finite element analysis shows that, for a nominal stress of 15 MPa, the maximum tensile stress at the interface is 178 MPa when a chisel is used for loading. With the wire loop, the maximum tensile stress is 69 MPa. The higher the stress concentration at the load application area, the lower the bond strength. Therefore, the use of a knife-edge chisel results in lower bond strength values than the wire loop, where the load is distributed over a larger area. Typical dentin bond strength values with macro-shear tests are 10 to 50 MPa. Cohesive and mixed failures are very frequent and may affect up to 55% of the specimens.

Another aspect of interest is the elastic modulus of the composite used for the specimen cylinder. The larger the mismatch between the elastic modulus of the composite and the elastic modulus of the substrate, the higher the stress concentration at the interface. This lowers the measured bond strength.

When measuring shear bond strength values, stresses at the interface are not uniformly distributed- Bond strength is reported as the nominal stress value (in MPa), that is, the failure load (in Newtons) divided by the entire bonded area (in mm^2). This is often not accurate because the stress distribution at the interface is very heterogeneous. Debonding occurs due to stress concentration around a critical size flaw, or void, at the interface that causes a crack to propagate. The actual stress level that initiates crack propagation can be several times higher than the nominal (or average) value. Therefore, nominal bond strength does not represent the failure stress.

Results of different studies are not comparable- Bond strength values for a specific material can vary a lot among studies due to differences in the bonding substrate, specimen preparation, storage conditions, and loading method. Unfortunately, there is very little standardization among research laboratories. Comparisons among different studies must be done very carefully.

Bond strength tests lack clinical significance- Based on what was described above, a threshold bond strength value that can be associated with a good clinical performance cannot be determined. Nevertheless, similar trends can be found in the literature for some adhesive systems. Systems that show poor performance in vitro generally have poor clinical performance.

Interfacial bond strength can be tested by a variety of methods. Using the dimensions of the bonded area, bond strength methods can be categorized as macro (4-28 mm^2) or micro (approximately 1 mm^2). The interface can be loaded either in tension or shear.

The ceramic-composite bond is susceptible to chemical,[77,78] thermal,[79] and mechanical[80] influences under intraoral conditions. The simulation of such influences in the laboratory is compulsory to draw conclusions on the long-term durability of a specific bonding procedure and to identify superior materials and techniques. Long-term water storage[81] and thermo- cycling of bonded specimens are accepted methods to simulate aging and to stress the bonding interface. Most studies that apply these methods reveal significant differences between early and late bond strength values.[82-86] Application of mechanical cyclic loading (fatigue load) causes significant reduction of bond strengths.[87,88]

Material selection and clinical recommendations on resin bonding to ceramics are based on mechanical lab- oratory tests that show great variability in materials and methods.[89,90] Preferred bond strength tests are the 3-point bending test, the tensile and micro-tensile test, and the shear and micro-shear test. Øilo[91] discussed the accuracy and clinical relevance of the different testing methods. The most common testing method is the shear bond test; however, some researchers prefer modified tensile tests to eliminate the occurrence of nonuniform interfacial stresses typical to conventional tensile and shear bond tests. Their specific fracture pattern may cause cohesive failure in the ceramic,[92] which may lead to erroneous interpretation of the actual data and taint an absolute ranking of the tested methods and materials.[89,90]

Research objective:

The purpose of this in vitro study was to evaluate the influence of different surface treatment methods on the resin bond durability to zirconia.

Hypothesis:

The surface treatment methods employed will positively influence bonding to zirconia. More specifically, the shear bond strength of resin cement to zirconia will be improved after surface modification with one or more agents both immediately and after thermal aging (thermocycling).

Materials and Methods:

Square-shaped samples of unprocessed zirconium-oxide ceramic (KATANA Zirconia STML, Kuraray Noritake, Japan) with the dimensions 12 mm × 12 mm × 3 mm were fabricated and then sintered in an induction furnace (CEREC Sintering furnace, Dentsply Sirona, Germany). N=20, Total N=100. Specimens were embedded in copper molds using PMMA (Polymethyl methacrylate) with one surface exposed for bonding. Specimens were ground finished with up to 800-grit silicon carbide abrasive under cooling water. Cylindrical composite resin specimens (2.1 mm diameter, 3 mm height) were fabricated using a standardized mold and packable composite resin material. Composite resin specimen surfaces were standardized using 800- grit silicon carbide abrasive with cooling water and cleaned in alcohol bath ultrasonically before cementation for 5 m, and thoroughly washed again with running distilled water. Specimens in Group 1 had no surface treatment, Group 2 was air- abraded with 50µm aluminum oxide at 2 bar pressure from a distance of 10 mm for 10 s, Group 3 was air abraded with glass bead particles of size 50µm at 0.25 MPa pressure from a distance of 10 mm for 20 s, Group 4 specimens were immersed in Zircos E etching solution for 2 h and Group 5 specimens were immersed in 48% Hydrofluoric acid solution heated at 25° C for 30 m. Cylindrical composite resin specimens (2.1 mm diameter, 3 mm height) were bonded to the zirconia surfaces with Dual cure resin cement (Panavia V5; Kuraray Noritake).


Figure 1

Figure 2

Figure 3

Figure 4

The test groups are:

Air abrasion using alumina particles; Henry Schein

Figure 5

Air abrasion using glass bead particles; Vaniman Manufacturing Co.

Figure 6

Zircos E solution; RH Marketing, Inc.

Figure 7

Contents: Nitric acid, hydrochloric acid, hydrofluoric acid, sulfuric acid and phosphoric acid.

48% Hydrofluoric acid solution; Sigma- Aldrich

Figure 8

Panavia V5; Kuraray Noritake (PV5):

Figure 9

Properties:

- Filler loading: 61 gew% (38 vol%)
- Flexural strength[1•2]: 127 MPa
- Flexural modulus[1•2]: 6,3 GPa
- Compressive strength[1]: 310 MPa
- Water sorption[1•2]: 21 $\mu g/mm^3$
- Film thickness[1•2]: 12 µm
- Radiopacity[2]: 180% Al
- Fluoride releasing (28 days)[1]: 58 µg/g
- Working time: (23˚C) 2 min.
- Curing time: (light) 10 sec.

[1] Dual curing of the paste (combination of self- and light-curing)
[2] According to ISO 4049:2009. Source: Kuraray Noritake Dental Inc.

Contents:

Paste A/Paste B
• Bisphenol A diglycidylmethacrylate (Bis-GMA)
• Triethyleneglycol dimethacrylate (TEGDMA)

• Hydrophobic aromatic dimethacrylate
• Hydrophilic aliphatic dimethacrylate
• Initiators
• Accelerators
• Silanated barium glass filler
• Silanated fluoroalminosilicate glass filler
• Colloidal silica Bisphenol A
• diglycidylmethacrylate (Bis-GMA)
• Hydrophobic aromatic dimethacrylate
• Hydrophilic aliphatic dimethacrylate
• Silanated barium glass filler
• Silanated alminium oxide filler
• Accelerators
• dl-Camphorquinone
• Pigments
CLEARFIL™ Ceramic Primer Plus
• 3-Methacryloxypropyl trimethoxysilane
• 10-Methacryloxypropyl dihydrogen phosphate (MDP)
• Ethanol
PANAVIA™ V5 Tooth Primer
• 10-Methacryloyloxydecyl dihydrogen phosphate (MDP)

• 2-Hydroxyethyl methacrylate (HEMA)
• Hydrophilic aliphatic dimethacrylate
• Accelerators
• Water

A load of 1000 g was applied for 10 m during the cementation process and light irradiated for 20 s from buccal, lingual, mesial, and distal sides for a total of 80 s.

Each main group was divided into two subgroups (n = 10/each). Half of the samples were tested for SBS after 48 h in distilled water at 37^{o} C (100-percent humidity), then subjected to 10,000 thermo cycles and the other half were tested after 10,000 thermo cycles.

The thermocycling was applied over 9 d, 30 s dwell time and 5 s in between baths. Specimens were placed in a fixture on a universal testing machine (Instron), aligned with the shearing blade just touching the bonding interface. A shear load was applied until failure at a crosshead speed of 0.5 mm/m. Loads were converted to MPa by dividing the failure load by the bonding surface area.

The fractured surfaces were inspected with a stereo microscope to evaluate the failure mode and were classified as adhesive, cohesive or mixed failures approximated by the amount of remaining resin cement on the ceramic surface in respect to the bonding surface area.

The data was analyzed by one-way ANOVA and paired t-test with $\alpha=0.05$.

Figure 10

Figure 11

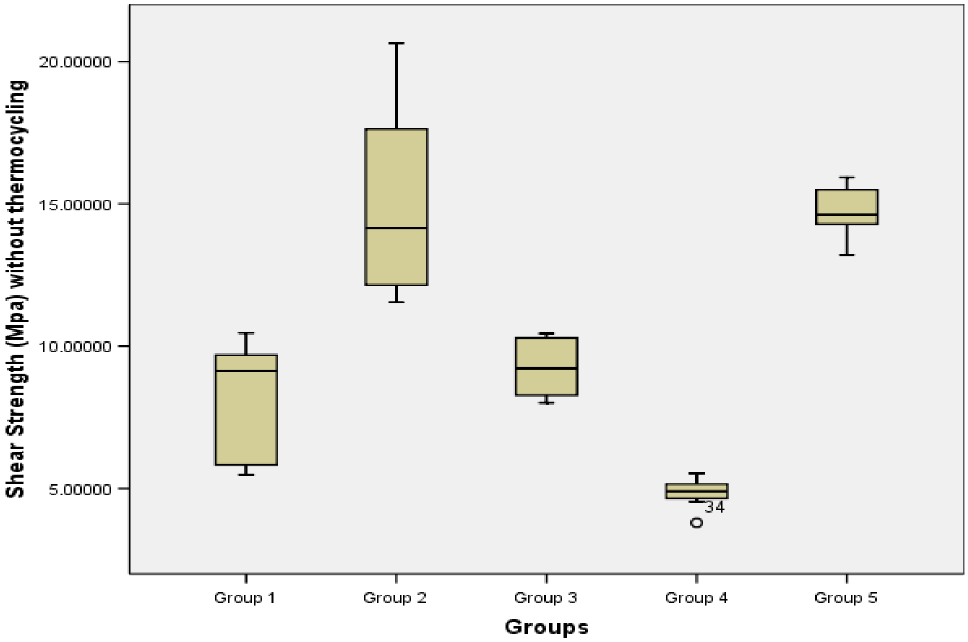
Results:

Table 1: Distribution of study population according to

	Shear strength [MPa] without thermocycling					
	Mean	Std. Deviation	95% CI		F-value	p-value
Group 1	8.316	1.953	6.919	9.713	60.572	0.001*
Group 2	14.976	3.189	12.695	17.257		
Group 3	9.286	0.985	8.582	9.991		
Group 4	4.831	0.468	4.497	5.166		
Group 5	14.796	0.829	14.203	15.390		

One-way ANOVA test *** Significant difference**

The mean Shear strength [MPa] without thermocycling was compared between group 1, group 2, group 3, group 4 and group 5 using the one-way ANOVA test. There was a significant difference in mean Shear strength [MPa] without thermocycling between group 1, group 2, group 3, group 4 and group 5.



Graph 1

Table 2: Distribution of study population according to

		Shear Strength without thermocycling			
		Mean Difference	p-value	95% CI	
Group 1	Group 2	-6.660	0.001*	-9.163	-3.757
Group 1	Group 3	-0.970	1.000	-1.145	4.261
Group 1	Group 4	3.485	0.011*	1.082	6.488
Group 1	Group 5	-6.480	0.001*	-1.164	4.242
Group 2	Group 3	5.689	0.001*	5.315	10.721
Group 2	Group 4	10.145	0.001*	7.541	12.948
Group 2	Group 5	0.179	1.000	5.296	10.702
Group 3	Group 4	4.455	0.004*	-0.476	4.930
Group 3	Group 5	-5.510	0.001*	-2.722	2.685
Group 4	Group 5	-9.965	0.001*	-4.948	0.458

Post-hoc bonferroni test *** Significant difference**

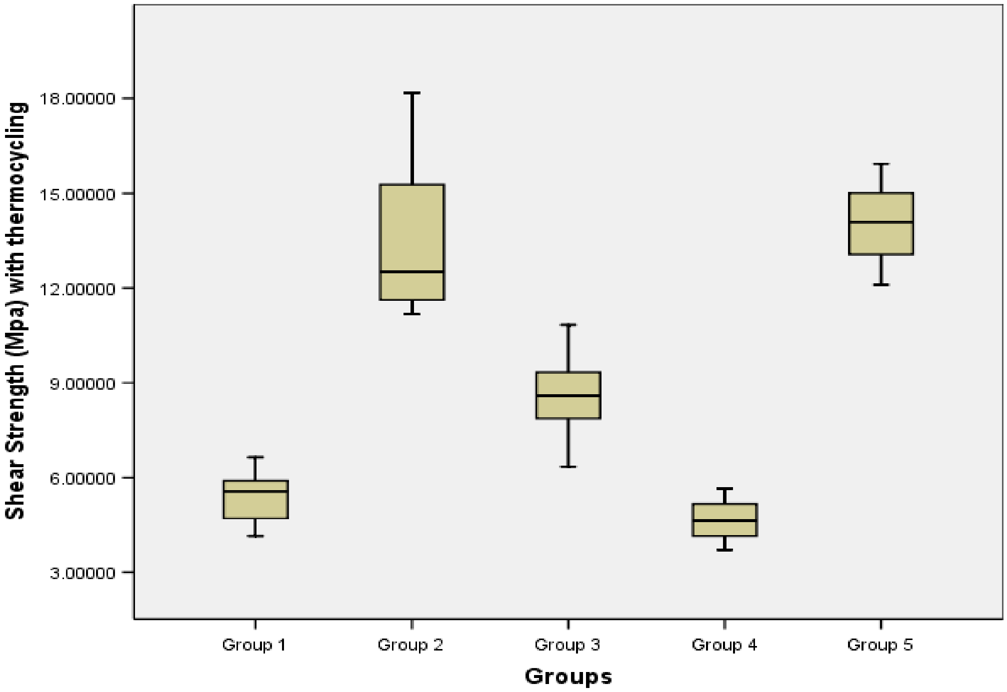
The inter-group comparison of mean Shear strength [MPa] without thermocycling was done using the post-hoc bonferroni test. The mean Shear strength [MPa] without thermocycling was significantly more among group 2 and group 5 compared to group 3 which was significantly more than group 1 and which was significantly more than group 4.

Table 3: Distribution of study population according to

	Shear strength [MPa] with thermocycling					
	Mean	Std. Deviation	95% CI		F-value	p-value
Group 1	5.446	0.803	4.872	6.021	95.770	0.001*
Group 2	13.466	2.328	11.800	15.131		
Group 3	8.556	1.349	7.591	9.521		
Group 4	4.160	0.627	4.212	5.109		
Group 5	13.146	1.274	13.035	14.857		

One-way ANOVA test *** Significant difference**

The mean Shear strength [MPa] with thermocycling was compared between group 1, group 2, group 3, group 4 and group 5 using the one-way ANOVA test. There was a significant difference in mean Shear strength [MPa] with thermocycling between group 1, group 2, group 3, group 4 and group 5.



Graph 2

Table 4: Distribution of study population according to

		Shear Strength without thermocycling			
		Mean Difference	p-value	95% CI	
Group 1	Group 2	-8.019	0.001*	-9.163	-3.757
Group 1	Group 3	-3.110	1.000	-1.145	4.261
Group 1	Group 4	0.786	0.010*	1.082	6.488
Group 1	Group 5	-8.100	0.001*	-1.164	4.242
Group 2	Group 3	4.910	0.009*	5.315	10.721

Group 2	Group 4	8.805	0.001*	7.541	12.948
Group 2	Group 5	-0.080	1.000	5.296	10.702
Group 3	Group 4	3.896	0.011*	-0.476	4.930
Group 3	Group 5	-4.990	0.001*	-2.722	2.685
Group 4	Group 5	-8.886	0.001*	-4.948	0.458

Post-hoc bonferroni test *** Significant difference**

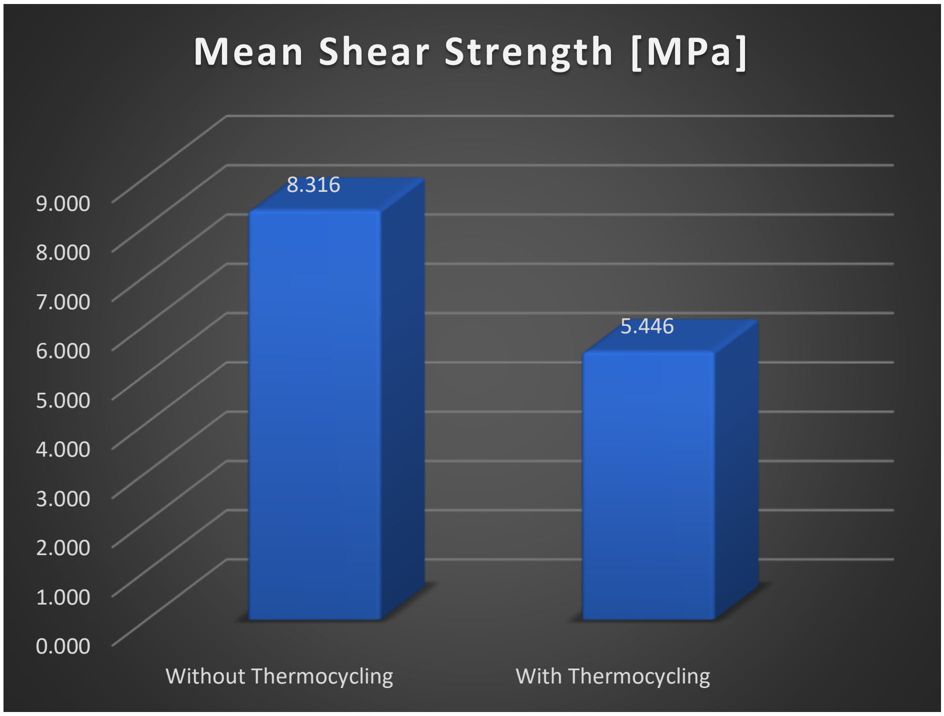
The inter-group comparison of mean Shear strength [MPa] with thermocycling was done using the post-hoc bonferroni test. The mean Shear strength [MPa] with thermocycling was significantly more among group 2 and group 5 compared to group 3 which was significantly more than group 1 which was significantly more than group 4.

Table 5: Distribution of study population according to

Group 1	Paired Differences				t-test value	p-value
	Mean	Std. Deviation	Mean	Std. Error Mean		
Without Thermocycling	8.316	1.953	2.870	0.517	5.556	0.000
With Thermocycling	5.446	0.803				

Paired t-test *** Significant difference**

The mean Shear strength [MPa] was compared between group 1 without thermocycling and with thermocycling using the paired t-test. The mean Shear strength [MPa] was significantly more among without thermocycling compared to with thermocycling.



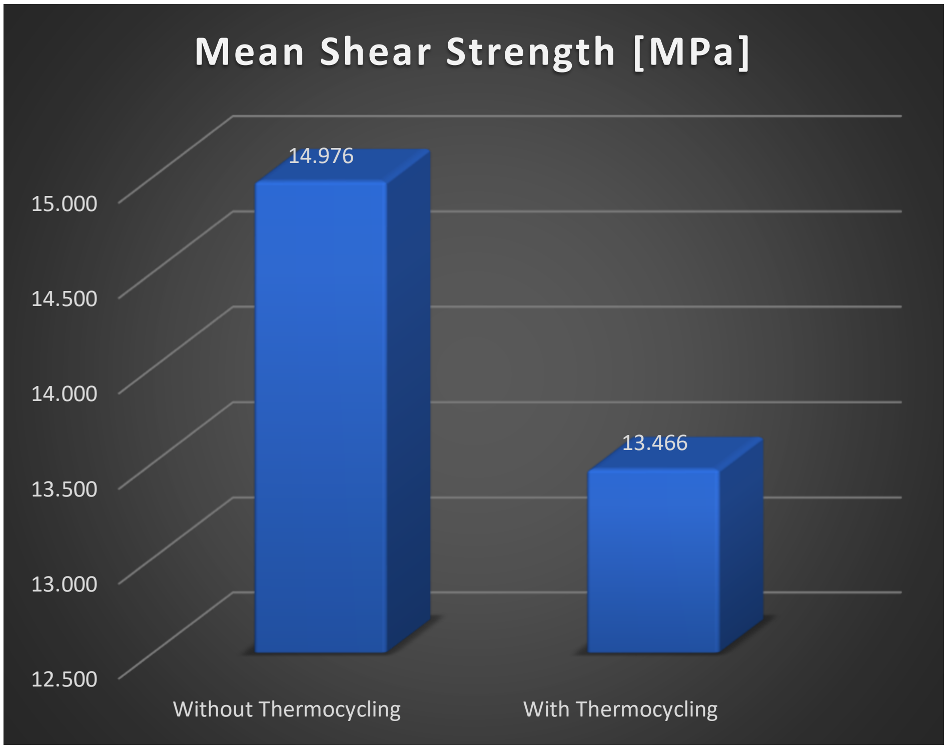
Graph 3

Table 6: Distribution of study population according to

Group 2	Paired Differences				t-test value	p-value
	Mean	Std. Deviation	Mean	Std. Error Mean		
Without Thermocycling	14.976	3.189	1.510	0.583	2.592	0.029*
With Thermocycling	13.466	2.328				

Paired t-test *** Significant difference**

The mean Shear strength [MPa] was compared between group 2 without thermocycling and with thermocycling using the paired t-test. The mean Shear strength [MPa] was significantly more among without thermocycling compared to with thermocycling.



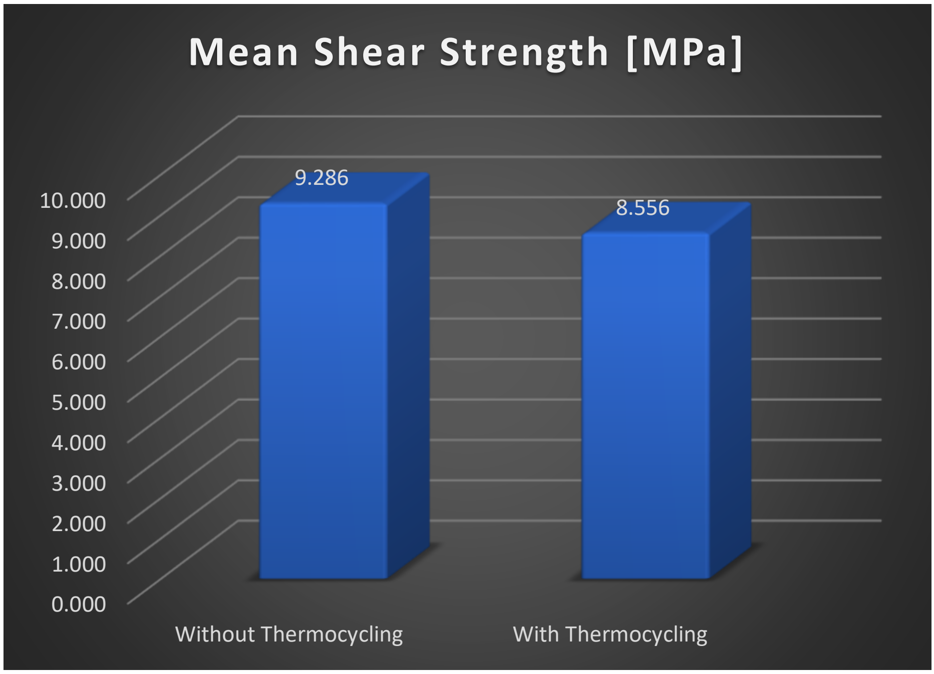
Graph 4

Table 7: Distribution of study population according to

Group 3	Paired Differences				t-test value	p-value
	Mean	Std. Deviation	Mean	Std. Error Mean		
Without Thermocycling	9.286	0.985	0.730	0.551	2.327	0.047*
With Thermocycling	8.556	1.349				

Paired t-test *** Significant difference**

The mean Shear strength [MPa] was compared between group 3 without thermocycling and with thermocycling using the paired t-test. The mean Shear strength [MPa] was significantly more among without thermocycling compared to with thermocycling.



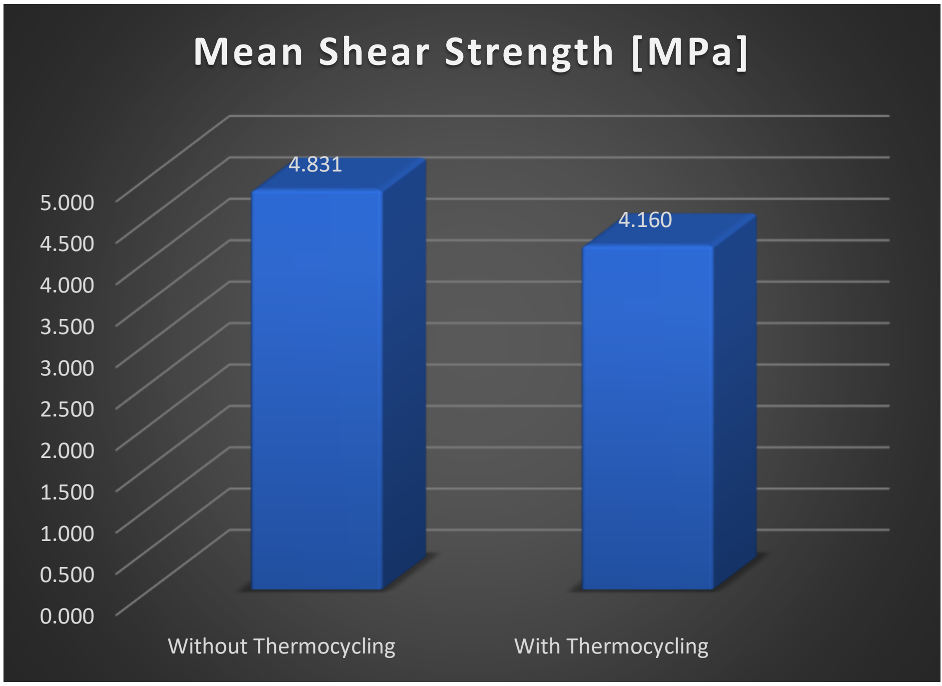
Graph 5

Table 8: Distribution of study population according to

Group 4	Paired Differences				t-test value	p-value
	Mean	Std. Deviation	Mean	Std. Error Mean		
Without Thermocycling	4.831	0.468	0.771	0.137	2.250	0.048*
With Thermocycling	4.160	0.627				

Paired t-test *** Significant difference**

The mean Shear strength [MPa] was compared between group 4 without thermocycling and with thermocycling using the paired t-test. The mean Shear strength [MPa] was significantly more among without thermocycling compared to with thermocycling.



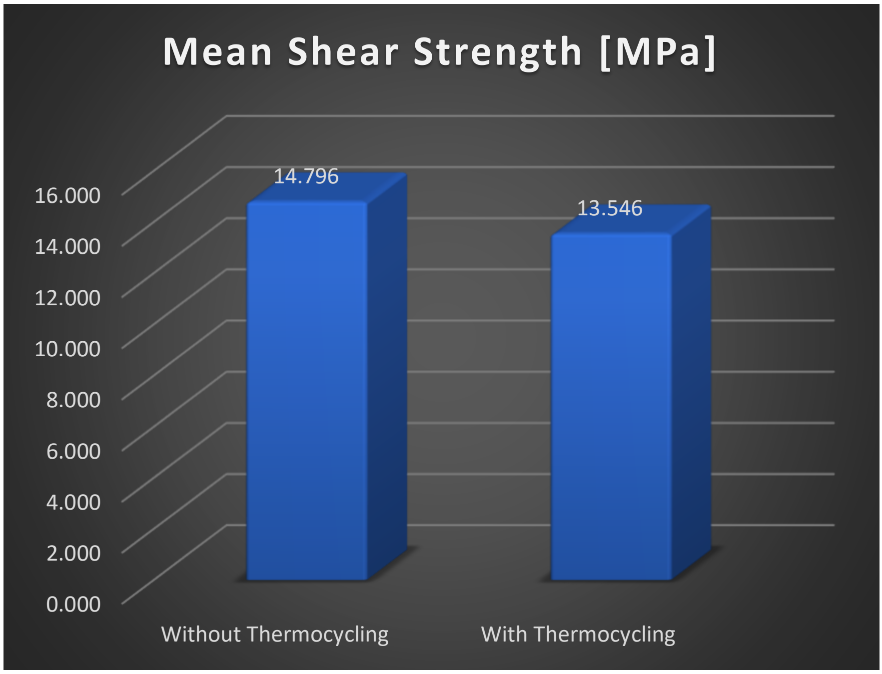
Graph 6

Table 9: Distribution of study population according to

Group 5	Paired Differences				t-test value	p-value
	Mean	Std. Deviation	Mean	Std. Error Mean		
Without Thermocycling	14.796	0.829	1.250	0.560	4.518	0.023*
With Thermocycling	13.546	1.274				

Paired t-test *** Significant difference**

The mean Shear strength [MPa] was compared between group 5 without thermocycling and with thermocycling using the paired t-test. The mean Shear strength [MPa] was significantly more among without thermocycling compared to with thermocycling.



Graph 7

The fractured surfaces were inspected with a stereo microscope to evaluate the failure mode and were classified as adhesive, cohesive or mixed failures approximated by the amount of remaining resin cement on the ceramic surface in respect to the bonding surface area.

A scanning electron microscope (FEI Quanta 600 ESEM; FEI Co) in the Singh Center for Nanotechnology at University of Pennsylvania was used to image and evaluate the failure modes.

Images of the specimens were captured at 75x and 500x magnification for detailed evaluation.

Majority of the specimens had a mixed failure mode and pictures from SEM evaluation can be noted here.

Figure 12

Scanning electron microscope

Group 1- No surface Treatment (Control group)

Sample shown below in figure 13 (Group1 Without Thermocycling) has mixed failure with 44% of the specimen showing cohesive failure within the resin and 56% showing adhesive failure exposing the zirconia surface.

All tested samples in this group showed mixed failures with less than 50% areas of cohesive failure within the resin.

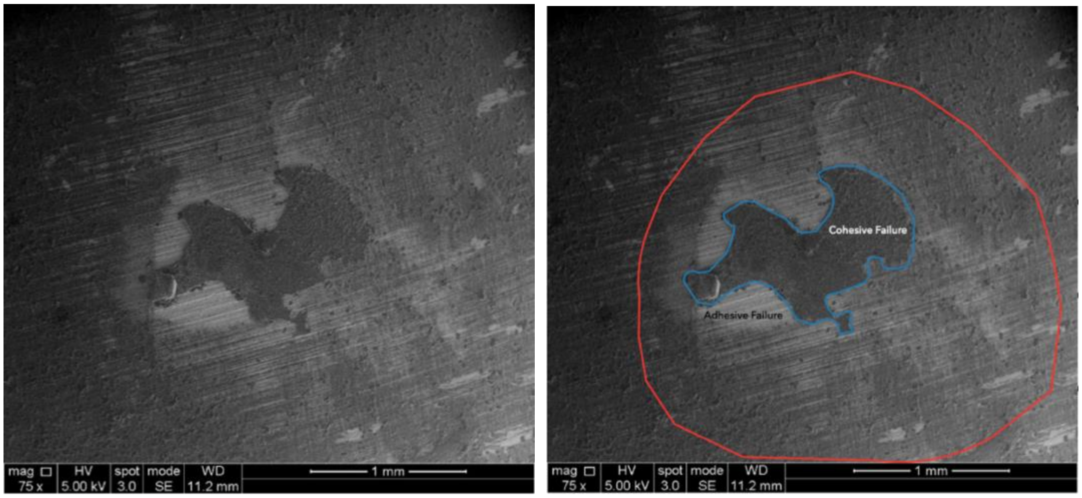


Figure 13

Sample shown below in figure 14 (Group1 With Thermocycling) has 100% adhesive failure exposing the zirconia surface.

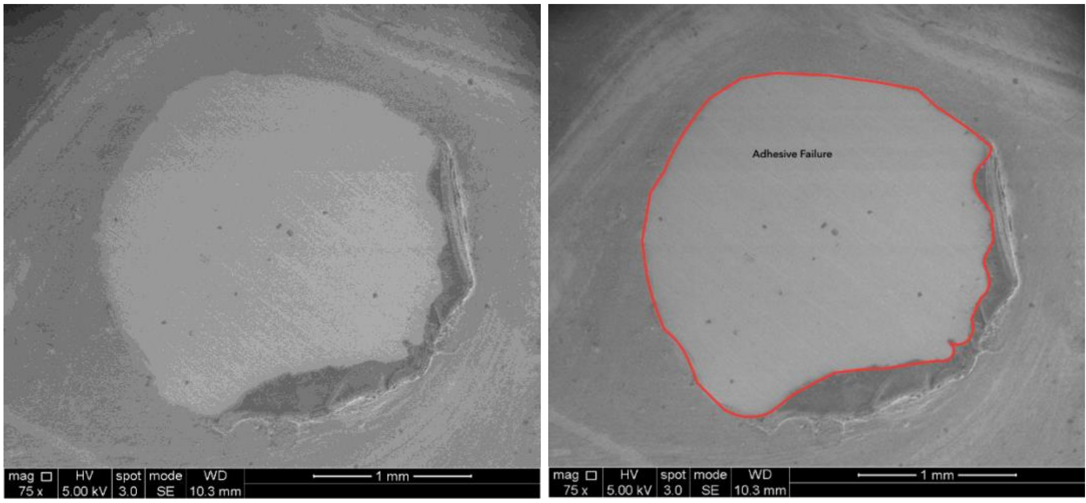


Figure 14

Group 2: Air abrasion using alumina particle

Sample shown below in figure 15 (Group 2 Without Thermocycling) has mixed failure with 37.5% of the specimen showing cohesive failure within the resin and the remaining surface showing resin infiltrates (62.5%) on zirconia surface (adhesive failure).

All tested samples showed mixed failures with variable amounts of cohesive and adhesive failures.

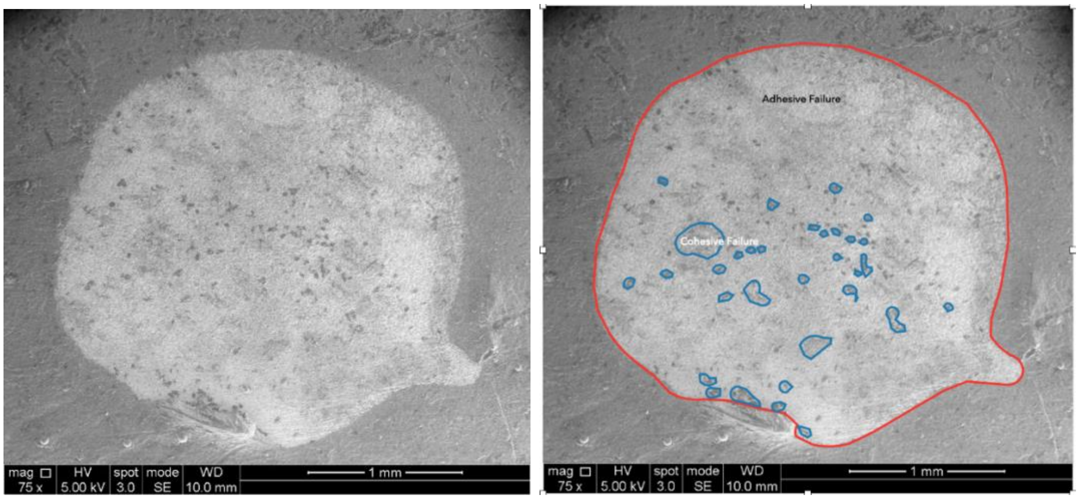


Figure 15

Sample shown below in figure 16 (Group 2 With Thermocycling) has mixed failure with 15% of the specimen showing cohesive failure within the resin and the remaining surface showing resin infiltrates on zirconia surface (mixed failure).

All tested samples showed mixed failures with variable amounts of cohesive and adhesive failures.

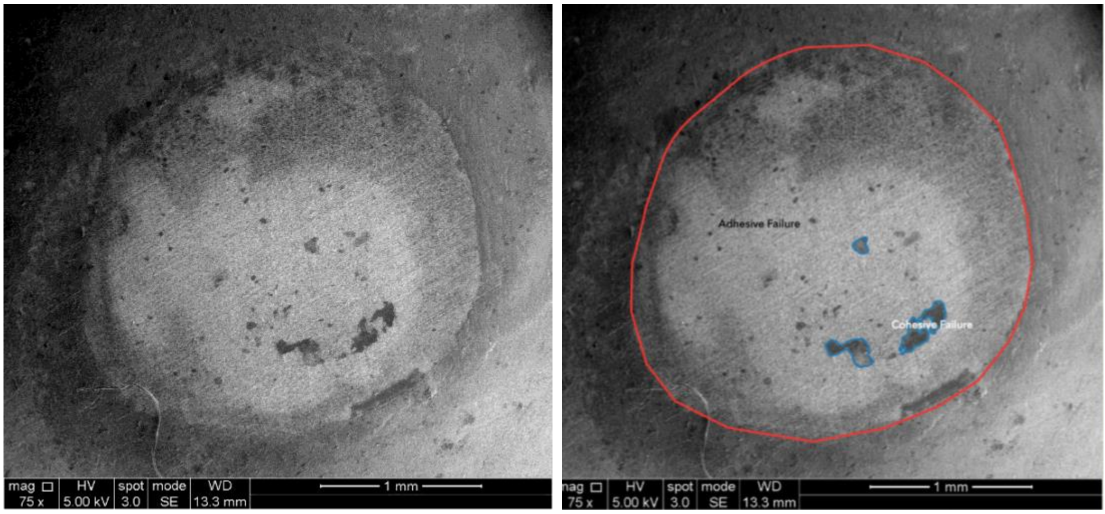


Figure 16

<u>Group 3: Air abrasion using glass bead particles</u>

Sample shown in figure 17 (Group 3 Without Thermocycling) has mixed failure with 66% of the specimen showing adhesive failure exposing the zirconia surface and 33% cohesive failure within the resin showing resin infiltrates on zirconia surface. All tested samples showed mixed failures with variable areas of cohesive and adhesive failures.

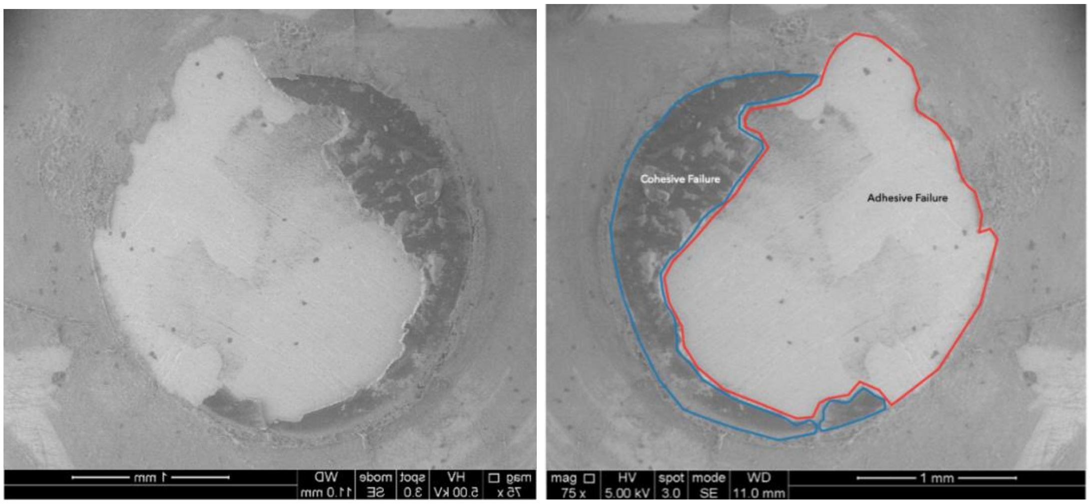


Figure 17

Sample shown below in figure 18 (Group 3 With Thermocycling) has mixed failure with 28% of the specimen showing cohesive failure within the resin and the remaining surface showing resin infiltrates on zirconia surface (adhesive failure).

All tested samples showed mixed failures with variable amounts of cohesive and adhesive failures.

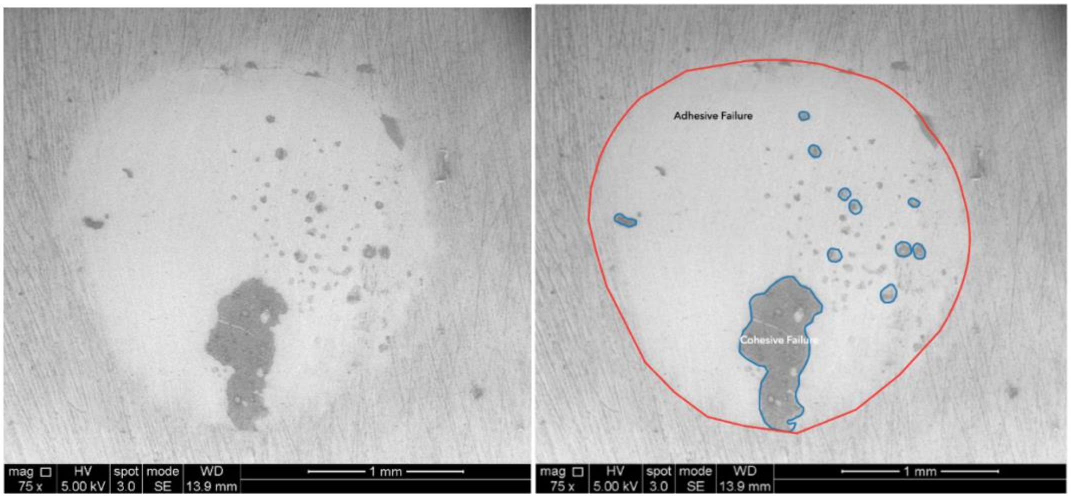


Figure 18

Group 4: Zircos E etching solution

Sample shown in figure 19 (Group 4 Without Thermocycling) has mixed failure with 25% of the specimen showing cohesive failure within the resin and 75% showing adhesive failure exposing the zirconia surface.

All tested samples showed mixed failures with variable amounts of adhesive and cohesive failure.

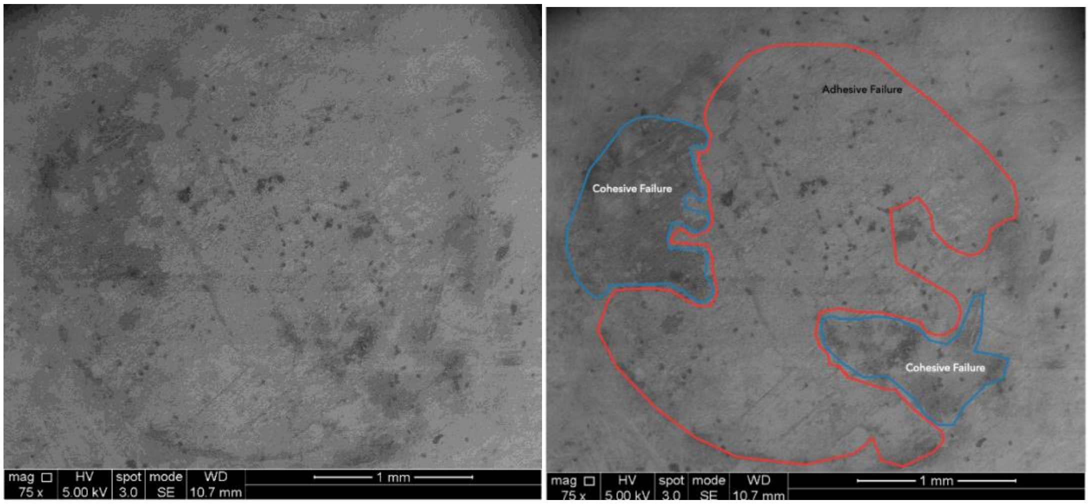


Figure 19

Sample shown below in figure 20 (Group 4 With Thermocycling) has 100% adhesive failure with remaining surface showing resin infiltrates on zirconia surface. All tested samples showed adhesive failures for this group.

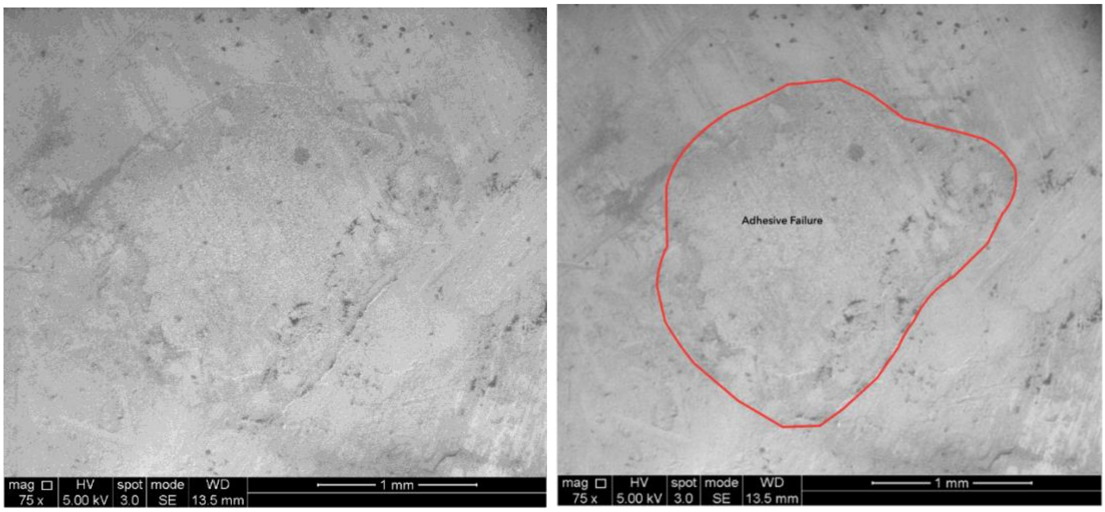


Figure 20

Group 5: Immersion in 48% Hydrofluoric acid solution

Sample shown in figure 21(Group 5 Without Thermocycling) has mixed failure with 21% of the specimen showing cohesive failure within the resin and 79% showing adhesive failure exposing the zirconia surface.

All tested samples showed mixed failures with variable amounts of adhesive and cohesive failure.

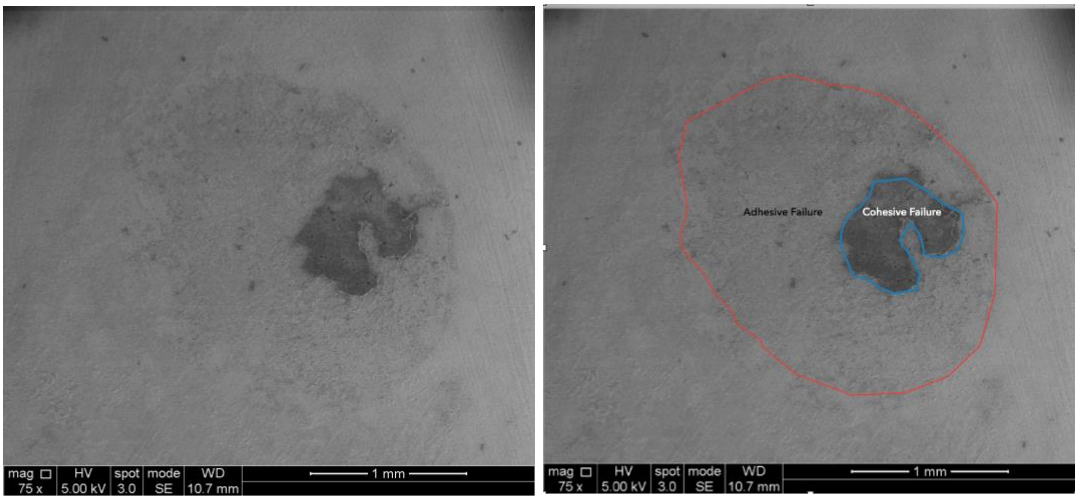


Figure 21

Sample shown below in figure 22 (Group 5 With Thermocycling) has mixed failure with 20% of the specimen showing cohesive failure within the resin and the remaining 80% of the surface showing resin infiltrates on zirconia surface (adhesive failure).

All tested samples showed mixed failures with variable amounts of cohesive and adhesive failures.

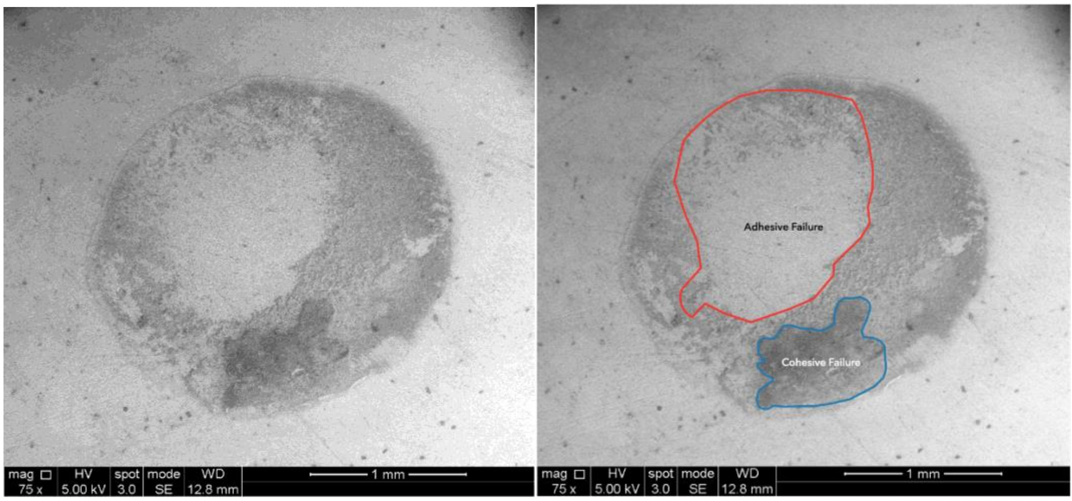


Figure 22

Discussion

The results of this study rejected our null hypotheses that various surface treatment methods will not influence bond strength values to zirconia. All the four surface treatment methods were significantly different from each other and with control group (no surface treatment). Based on the results, it can be inferred that choosing correct surface treatment method is imperative for achieving optimal bond strength of zirconia. Air abrasion and heated hydrofluoric acid proved to be better methods for surface treatment of zirconia as compared to other methods. Earlier studies have suggested that hydrofluoric acid does not etch zirconia because of the absence of glassy phase, however, results from the study suggest that hydrofluoric acid when heated etches zirconia and improves bond strength and is comparable to air abrasion. In terms of clinical implications, air abrasion still can be vouched to be a safer method for surface modification of zirconia as utmost care is required for using hydrofluoric acid as a surface modification method.

The surface treatment methods used in the present study have been used by various authors in the past, but results are contraindicating. Also, these methods have never been assessed together in a single study. Surface modification of zirconia using air abrasion has been gold standard. Air abrasion using alumina particles and glass beads have been used and compared by various authors. Airborne-particle abrasion with Al_2O_3 combined with the use of MDP primer with resin cement has been recommended for bonding a zirconia crown due to the formation of a durable zirconia-resin bond.[70,93-95] Airborne-particle abrasion may, however, create microcracks on the surface of zirconia and reduce the fracture resistance of the restorations[75,96], therefore, solutions for etching zirconia have been developed, including a solution composed of multiple acids, which can increase zirconia's surface roughness.[75] The use of this acidic solution has been reported to increase the bond strength of resin cement to zirconia.[75,97] The zirconia etching solution contained hydrofluoric acid (HF), hydrochloric acid (HCl), sulfuric acid (H_2SO_4), nitric acid (HNO_3), and phosphoric acid (H_3PO_4). Also, surface modification by HF has always been controversial because of absence of glassy phase in zirconia. However, Sriamporn (2014)[73] concluded that at higher concentration, heated HF can etch dental zirconia ceramic, creating micro-morphological changes. Cavitations were found on zirconia surfaces in SEM images taken after surface conditioning using 9.5% hydrofluoric acid at room temperature for 1, 2, 3, and 24 h, in 80°C for 1, 3, 5, and 30 m, and using 48% hydrofluoric acid for 30 and 60 m. Moreover, a mix of tetragonal form and monoclinic form were observed after surface conditioning when compared to the predominant tetragonal form prior to surface conditioning.

The most common test method to assess bonding effectiveness to zirconia is the shear bond-strength test [26], most likely because it requires nearly no further specimen processing of the fully sintered zirconia, once the bonding procedure is completed. However, the shear-bond strength test has been repeatedly documented to result in inhomogeneous stress distribution along the interface, for instance often leading rather to 'cohesive' failures in the substrate than to 'adhesive' failures at the actual interface.[26,90]

Conventional shear bond and tensile tests are limited by specific fracture patterns, which may cause cohesive failure in the ceramic or composite. The fractures seen in the present study are predominantly mixed or cohesive within the composite resin. This failure indicates that the bond strength exceeds the fracture resistance of the composite resin material, which limits the ability to interpret the acquired data since fracture strength rather than the bond strength is tested in those instances. In the present study, there were no cohesive failures in zirconia ceramic since the fracture strength of zirconia ceramic far exceeds that of the composite resin and the bond strength.

In the present study, the bonding of the specimens was achieved under 1000g load to achieve homogenous film thickness and uniform interface. Majority of the specimens had mixed failures with part of the specimen showing adhesive failure and part of the specimen showing cohesive failure within composite.

While in vitro studies indicate a significant increase in flexural strength of high-strength ceramic restorations after resin bonding[98], the evidence on the exact influence of the cementation medium on clinical performance is limited. For full-coverage high-strength ceramic crowns and FDPs and based on the specific clinical situation, the clinician can choose between resin bonding with composite resins, insertion with a self-adhesive resin, or conventional cementation with zinc-phosphate, glass ionomer, or resin-modified glass ionomer cement.[19]

In the mouth, water and repeated thermal changes constantly degrade and hydrolyze the ceramic/composite-resin interface. Long-term water storage at a constant temperature or thermal cycling are the most often used conditions to simulate aging of resin bonds. However, it has been shown that different bonding systems are influenced differently by these two parameters.[26]

Multiple studies have shown the effect of simulated aging on the resin-ceramic bond.[6,17,18] Even though direct clinical correlations and conclusions cannot be drawn from such ageing methods, their significant effects on the bonding interface have been demonstrated by multiple studies. Long term storage and thermocycling dramatically decreased resin bonds to high strength ceramic materials, underlining the need for such methods in order to enhance clinical relevance. Studies have employed short term water storage for 3 days [18,99], short term thermocycling of 10,000 cycles[100] or long term thermocycling of 12,000 cycles over 180 d [18,99]. In the present study, bonded specimens were stored at 37^{o} C for 48 h before short term thermocycling of 10,000 cycles over 10 d between 5^{o} C and 55^{o} C with a dwell time of 30 s. Although it is difficult to calculate the amount of clinical ageing associated with amount of simulated ageing, it is important to incorporate some form of simulated ageing before testing bond strength to enhance clinical relevance.

The results of the present study conclude that mean shear bond strength both with and without thermocycling was significantly more among group 2 (air abrasion using alumina particle) and group 5 (48% HF solution heated at 25^{o} C) compared to group 3 (air abrasion using glass bead particles) which was significantly more than group 1(no surface treatment) which in turn was significantly more than group 4 (Zircos E etching solution). The comparable mean shear bond strength for group 2 and group

5 is in accordance with the work conducted by Zhang Q et al (2020) where they compared shear bond strength of zirconia samples with surface treatment by HF and sandblasting. They concluded that shear bond strength of samples etched with HF was comparable to the samples that were sandblasted. HF etching creates a nano roughness surface that significantly increases SBS and surface hydrophilicity, with the minimal damage to zirconia. Therefore, HF etching is a promising method for conditioning zirconia surfaces. Not only the roughness height, but also the roughness width value, as well as frequency and regularity affect the bonding strength. Jin et al (2021)[101], in their study also found that the effect of HF acid etching on the bonding performance of ceramic-coated zirconia is concentration- and time-dependent. Their results showed that pre-treatment by acid etching with 9.5% HF for 2 m and 5% HF for 5 m resulted in the highest bond strengths, for both the ceramic-coating method as well as the conventional method ie, alumina air-abrasion combined with 10-MDP-containing primer. Harb O (2021)[102] also showed that hot acid etching pre-treatment improves the retention of resin cement to zirconia crowns. The use of MDP self-adhesive resin cement (Panavia SA Cement Plus) with hot acid etching is effective and can be used for adhesive cementation of zirconia crowns.

According to the mentioned results, air abrasion with alumina particles has better impact on the mean shear bond strength as compared to air abrasion with glass beads. This observation is similar to the study by Khanlar LN et al (2021)[58]. Mehari K

(2020)[103] also conducted research to evaluate the effects of air abrasion with aluminum oxide or glass beads to three types of zirconia containing various levels of cubic crystalline phases (3Y-TZP, Katana ML; 4Y-PSZ, Katana STML; and 5Y-PSZ, Katana UTML, Noritake) on the shear bond strength of resin cement. They concluded that a significant difference in shear bond strength was found based on the surface treatment ($p < 0.001$), but not on the type of zirconia. Also, air abrasion with glass beads or no surface treatment resulted in significantly lower bond strength of the resin cement to all three zirconia types compared to air abrasion with aluminum oxide. A systematic review conducted by Alammar A and Blatz M (2022)[93] also stated that bond strength values achieved with alumina air particle abrasion were over twice as high than with other treatment methods such as glass bead-air particle abrasion or no surface pretreatment. Al Mutairi R (2021)[104] too recommended use of alumina over glass beads for air abrasion of zirconia. They recommended use of smaller particle size for surface modification of zirconia to achieve evenly corroded surface topography. McLaren EA (2021)[105] also checked for biaxial flexural strength and surface topography following surface treatment by alumina and glass beads. He concluded that 4 mol% yttria-stabilized zirconia was stronger when alumina abraded than when glass bead abraded. Scanning electron microscopy showed that the surfaces airborne-particle abraded with glass beads displayed little difference; and those airborne particles abraded with alumina were the roughest.

The statistical analysis of the present study shows that mean shear bond strength for group 4 (Zircos E etching solution) was significantly lower than groups. Similar results were obtained in a study conducted by Sadid-Zadeh et al (2021)[106] where the mean shear bond strengths of resin cement to zirconia was the lower for Zircos E etching solution as compared to air particle abrasion. However, a significantly higher shear bond strength was observed in the specimen group treated with acid etching followed by air abrasion. Visual comparison of the SEM images in their study did not show a distinct difference in topography between the Zirconia etching solution and air abrasion groups. Cho JH (2017)[75] compared the shear bond strength to zirconia of different resin cements after surface treatment with Zircos E etching solution, air abrasion, and tribochemical silicacoating. Their results also showed that when Panavia was used as a luting media, the mean shear bond strength was more in the samples which were treated with air abrasion than those that were treated with Zircos E etching solution. The remarkably lower bond strength of Panavia F 2.0 in Zircos E etching system group shown in this study could be considered to be a consequence of the chemical bonding process. The MDP contained in Panavia F 2.0 reacts with hydroxyl group on the surface of zirconia. However, previous reports have been made that such reaction cannot be effectively retained when themocycled.[60,107] It could be hypothesized that thermocycling of zirconia could have eliminated the hydroxyl group on its surface, and hence reduced the shear bond strength of Panavia F 2.0.

Sales A et al (2022) [108] studied the effect of air abrasion, etching using Zircos E etching solution and a combination of these two surface treatment methods on the micro-shear bond strength and surface characteristics of translucent and opaque zirconia. Their results showed a higher shear bond strength value for zirconia treated with both air abrasion and etchant as well as the etchant alone as compared to air abrasion method. Conflicting results to the present study could be because of the use of different luting agent (RelyX Ultimate, 3M ESPE, St. Paul, Minnesota, USA) instead of Panavia V5 which was used in the present study. Also, the sample size was two samples for each group, this is acknowledged as the limitation of the research.

The result of the present study reveals that mean shear bond strength values for each group decreases after thermocycling. Since Panavia V5 has been used as the luting cement in the present study, it has been hypothesized that post-thermocycling, the bond strength to zirconia reduces as a result of hydrolysis of the chemical bonds between the primer and the cement. This in turn is because of hydrophilic unreacted silane molecules present in the primer. Similar observations were seen in a study conducted by Khanlar et al (2021) [58] where zirconia samples treated with glass beads could not survive thermocycling and the non-thermocycled group showed good bond strength when used along with an MDP-silane primer. Zirconia treated with alumina particle- air abrasion showed higher bond strength when used along with an MDP primer alone due to presence of long carbonyl chains that facilitated the formation of water-resistant chemical bond to zirconia. Lee et al (2019) [109] also found that use of self-adhesive resin cements (RelyX) resulted in a higher micro-tensile bond strength when compared to a self-etching resin cement (Panavia) after thermocycling. This is because self-adhesive cement penetrates more easily through gaps in the roughened surface to form microchemical interlocks and because the inorganic filler of the self-adhesive resin cement is more resistant to hydrolysis and plays an important role in cement formation.

Another reason for decreased bond strength post thermocycling can be that thermocycling may induce stress on the bonded interface due to different coefficients of thermal expansion of substrate and test materials. This could be attributed to two important factors; it might be due to degradation of the luting cement itself and the hydrolytic effect of water at the luting cement/ceramic interface due to thermal expansion of the bonded specimens which could result in hoop stress during thermocycling.[110]

The SEM images of the fractures seen in the present study are predominantly mixed or cohesive within the composite resin. This failure indicates that the bond strength exceeds the fracture resistance of the composite resin material, which limits the ability to interpret the acquired data since fracture strength rather than the bond strength is tested in those instances. In the present study, there were no cohesive failures in zirconia ceramic since the fracture strength of zirconia ceramic far exceeds that of the composite resin and the bond strength.

In the present study, the bonding of the specimens was achieved under 1000g load to achieve homogenous film thickness and uniform interface. Majority of the specimens had mixed failures with part of the specimen showing adhesive failure and part of the specimen showing cohesive failure within composite.

The SEM analysis showed an adhesive failure in samples surface treated with Zircos E solution while a mixed mode of failure were observed in the remaining groups. 100% adhesive failure in thermocycled samples treated with Zircos E clearly indicate its limited role in etching of zirconia. Adhesive failure can be attributed to poor bonding between the zirconia and cement due to cement deterioration as a result of hydrolysis of the bonds from thermocycling. Similar results were seen in the study conducted by Harb O et al [101] where the samples abraded by alumina particles and cemented by Panavia SA Cement Plus, showed a mixed mode of failure. Adhesive failure, which can be attributed to poor bonding between the zirconia and cement due to cement deterioration from thermocycling could be a cause of the failure. Mehari K et al (2020)[102] compared surface treatment of zirconia by air abrasion with alumina particles and glass beads. A higher bond strength, almost twice that in glass bead specimens, was seen with alumina particle specimens. A systematic review of the failure mode analysis also observed a mixed mode of failure more commonly seen in the alumina air abraded specimens than glass bead abraded specimens.[93] Mixed failure modes are often associated with higher bond strength compared to purely adhesive-type failures.[111]

Limitations of this study include the in-vitro set up, short term thermocycling application, bonding to resin vs. tooth structure, copper cups interacting with strong acids as well as being single-operator study. Other factors besides bond strength also play a role in success of full coverage and partial coverage zirconia restorations. Clinical studies with multiple operators and long-term performance in the oral environment can provide more clinically relevant data.

Conclusion: From the results of the present study, it can be concluded that air abrasion with alumina is the gold standard for surface treatment of zirconia before cementation. Heated HF at higher concentration definitely etches zirconia and is comparable to surface treatment with alumina. Zircos E though being combination of strong acids failed to show promising results. It can be hypothesized from this fact that heated strong acids can etch zirconia surface and hence increasing the bond strength of zirconia.

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