Original Article

Effect of Acidulated Phosphate Fluoride Gel on Zirconia Intaglio Surface: An *In-Vitro* Study

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ABSTRACT

Aim: To evaluate the micro-shear bond strength (μ-SBS) of resin-modified glass ionomer cement and to assess the chemical and topographical changes in the zirconia fitting surface induced by acidulated phosphate fluoride (APF) gel using scanning electron microscope (SEM) analysis and Fourier transform infrared (FTIR) spectroscopy. **Materials and Methods:** Thirty-two samples were prepared from two zirconia materials, UPCERA HT White and BruxZir® Solid Zirconia, milled by a computer-aided design/computer-aided manufacturing system. From each zirconia sample, six plates were prepared for FTIR and SEM testing. Following sintering, the samples were divided into control and test groups for each material. The APF gel (1.23%) was applied to the intaglio surface of each test group. To measure the μ-SBS between the zirconia materials and luting cement, 20 rectangular samples of zirconia material were prepared. Ten samples were obtained from Upcera and ten from Bruxzir, with five assigned to the control and five to APF groups. **Statistical Analysis:** For the μ-SBS test, independent samples *t* test was conducted to determine the level of significance between the tested groups. **Results:** FTIR spectroscopy revealed new bands for Upcera and Bruxzir zirconia owing to ion exchange between the formed sodium phosphate and the zirconia surface and the formation of zirconium phosphate by an ester reaction. SEM assessment identified lines, scratches, or surface dissociation that appeared on the intaglio–zirconia surface after conditioning. The μ-SBS test, as indicated by the independent samples *t* test, showed a significant increase in bond strength of 1.266 and 1.566 MPa for Upcera and Bruxzir zirconia, respectively. **Conclusion:** This study offers new practical, cost-effective, and accurate tests to enhance the μ-SBS of luting cement to yttria-stabilized tetragonal zirconia polycrystal ceramics.

Keywords: *Acidulated phosphate fluoride gel, Fourier transform infrared spectroscopy, micro-shear bond strength, zirconia*

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Introduction

High-strength zirconia ceramics are becoming increasingly popular in dentistry due to their superior mechanical properties, including high flexural strength, toughness, esthetic appeal, and

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biocompatibility. Moreover, the polycrystalline structure of zirconia ceramic makes it acid-resistant.[1,2]

Silica-based ceramics undergo etching with hydrofluoric (HF) acid following salinization to achieve robust bonding during cementation. In contrast, zirconia is a ceramic material free of silica and is unaffected by common etching methods. The zirconia surface is roughened by surface treatments, including air abrasion with aluminum oxide particles, thus enhancing the bond strength.[2,3] Additional zirconia surface treatments include laser treatment, hot etching solution, and selective infiltration etching (SIE).[4]

Alumina sandblasting of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), even at a high air pressure, cannot produce the same degree of surface roughness similar to silica-based ceramics because the hardness of Y-TZP is substantially higher than that of silica-based ceramics. Y-TZP has also been subjected to other surface roughening techniques, such as bur grinding, laser irradiation, hot acid etching, sintering, and SIE; however, these procedures require sophisticated methods and/or cause substantial Y-TZ phase changes.[5]

An ongoing debate exists regarding the ideal surface modifications to achieve the strongest bond between cement and zirconia despite numerous techniques and studies aimed at enhancing cement adhesion to zirconia.[6]

Various chemical agents can be used to bond the zirconia surfaces chemically. According to studies, strengthening and preserving the Y-TZP composite bond require chemical rather than physical processes.[7]

To enhance the micro-interlocking of silica-based ceramics, etching with a low concentration of HF acid at room temperature for a short duration (often several minutes or less) is frequently used in clinical settings. However, zirconia-based ceramics have not been roughened using this technique in clinical settings. Moreover, lower HF acid concentrations cannot quickly etch Y-TZP; therefore, reducing the exposure duration is preferred for clinical purposes. The mechanical characteristics of Y-TZP are harmed by prolonged immersion in 40% HF. Furthermore, high HF concentrations (40% and 48%) are too high for safe clinical application.[5,8]

Acidulated phosphate fluoride (APF) gel, frequently used to apply fluoride in office settings, contains sodium fluoride, phosphoric acid, and HF acid. The APF gel has been suggested as an alternative to HF acid for ceramic surface etching before bonding with composite resins. Unlike HF acid, which can potentially cause tissue rashes and burns, APF gel is considered safe for application on oral tissues.[9,10]

Zirconia treated with APF undergoes higher surface dissolution as ions move from the gel to the surface. However, the study did not observe phase changes in the crystal structure, indicating that the surface deterioration was limited to the superficial layers and did not cause aging.[11]

Fourier transform infrared (FTIR) spectroscopy allows the rapid differentiation of typical dental materials prepared from organic components for inorganic restorations. Scanning electron microscope (SEM) analyses enable the observation of the topography details of the surfaces.[12]

Shear bond strength (SBS) tests are more straightforward, predictable, and do not require additional preparation of the specimens before testing, compared to tensile bond strength tests, which are more difficult to prepare and align in the testing machine without creating a harmful stress distribution.[13,14]

This study examines how two commercially available partially stabilized zirconia ceramics react to APF treatment regarding their surface topography, chemical crystal structure, and micro-SBS (μ-SBS) of resin-modified glass ionomer cement/zirconia adhesion. The null hypotheses are as follows: (1) no chemical alterations would be evident on the treated zirconia surface using FTIR spectroscopy, (2) APF gel would not alter the topography of the zirconia surface, and (3) zirconia adhesion and μ-SBS in the context of resin-modified glass ionomer cement and zirconia adhesion would not exhibit improvement.

Materials and Methods

Two zirconia framework blanks (UPCERA HT White, China [UH]; BruxZir® Solid Zirconia, USA [BC]) were used in the study, and AutoCAD computer software was used to obtain the (*.STL) file of standard 3D geometrical design to prepare 32 samples (6 samples for FTIR, 6 samples for SEM, and the remaining 20 samples for μ-SBS) using the computer-aided design/computer-aided manufacturing (CAD/CAM) system, and Figure 1 shows the graphical representation for the samples construction and tests conducted in the study. Six zirconia plates (12 mm × 4 mm × 1 mm) were prepared and milled from the zirconia blocks for FTIR spectroscopic examination, with three samples allocated to each zirconia type. The specimens were carefully removed from the zirconia blanks after the milling procedure was complete, and the borders were adjusted with football-shaped fine-fissure

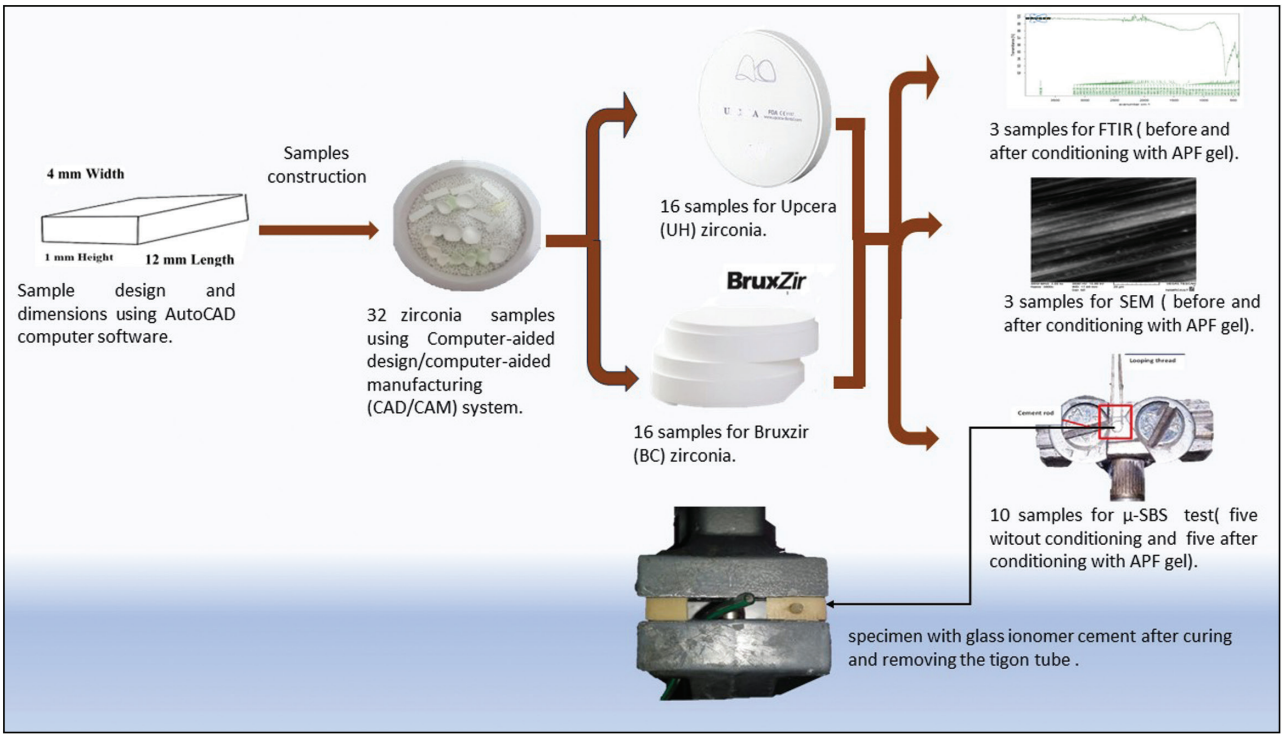


Figure 1: Graphical representation for the sample construction and tests conducted in the study

diamond burs (379-023M-HP, NTI-Kahla GmbH rotary instruments, Germany) and sintered in a furnace (MIHM-VOGT GmbH & Co. KG, Stutensee-Blankenloch, Germany). The outer contour surfaces of the prepared specimens were finely ground, polished, cleaned, dried, and glazed to replicate the externally finished surface of the extra coronal restoration and remove any signs of flaws and imperfections on that surface by using dental loupes. Before conditioning with APF1.23% (APF Fluoride Gel, USA; Deepak Products LLC), an FTIR spectrophotometer (ALPHA, Bruker, Germany) was used to measure the vibrations of the bonds and determine the structural, chemical, and band conditions of the zirconia material. These readings served as controls before the zirconia material was chemically conditioned. Subsequently, APF (1.23%) was applied for 10 min as the zirconia conditioning gel (APF group). Furthermore, all specimens were ultrasonically cleaned for 15 min and examined again for FTIR analysis.

For SEM analysis, another six similarly prepared zirconia specimens were categorized into groups, as previously described. The intaglio surface of these specimens was then treated in the same way as previously mentioned, and they were coated with gold–palladium and examined using the SEM at 15.00 kV electrical voltage and 3000× magnification power to observe the changes in their ultra-structure with an SEM (TESCAN, USA).

All these processes were conducted under the supervision of specialized experts for each part of the study.

To measure the μ-SBS between the zirconia materials and the resin-modified glass ionomer cement (RelyXTM luting 2; 3M ESPE, USA), 20 rectangular specimens (12 mm × 4 mm × 1 mm) of the zirconia material were prepared, with ten specimens allocated to each of UH and BC.

During cement application, a tigon (diameter: 1 mm and height: 1 mm) was placed at the center of the specimen intaglio surface and fixed in its place with the aid of an orthodontic wax, and the work was carried out. The chemical conditioning of the specimen intaglio surface was performed within the hole of the tigon tube. All the specimens were ultrasonically cleaned for 15 min before conditioning and tested using an ultrasonic cleaner (Bio Sonic® UC50DB) and gently dried with oil-free air for 1 min. For five specimens of each zirconia material type used in this study, the hole of the tigon tube was left unaltered (control group). In contrast, the other five specimens of each zirconia material type were conditioned using a disposable dental applicator (APF groups). The tigon tube area for the control and APF groups was then filled with resin-modified glass ionomer cement (RelyXTM luting 2; 3M ESPE, USA. After dispensing and mixing, the cement was gently applied, covered with a celluloid strip, and cured using a light-emitting diode device for 20 s. The specimens

were placed in an incubator at 37°C and immersed in distilled water for 24 h.[15]

With the help of two screws, the zirconia specimen was positioned and secured within a slot in a metal bar. The metal bar was welded to a rod, which was attached to the universal test machine's base via magnetic force [Figure 1].

Each specimen was subjected to a shear force at a rate of 0.5 mm/min until failure by using a universal test machine (Sans Testing Machine Co. Ltd. Shenzhen, China). To achieve the correct orientation for the shear test, the interface between the resin-modified glass ionomer and zirconia, thread loop, and load cell center was positioned straightforwardly [Figure 1][16,17]

The micro-shear value was calculated using Equation 1. As indicated below, stress can be equivalent to the force applied per cross-sectional area or the load per unit area.[14]

$$\tau = \frac{F}{A} N/mm^2 \quad (1)$$

τ = the shear stress (N/mm^2 or MPa), F = the force applied (N), and A = the cross-sectional area of the material with area (mm^2) parallel to the applied force vector.

Statistical analysis

The μ-SBS results obtained in this study were statistically analyzed using Statistical Package for Social Sciences version 27.0 (IBM Corp., Armonk, NY, USA). The data were statistically analyzed using a parametric independent one-sample *t* test at a confidence level of 95% to compare the effect of each variable and the statistical differences between the groups along with their confidence interval and effect size values.

Results

1. Fourier transform infrared spectroscopy test

For chemical structure evaluation, Figure 2a and b shows the IR chart of the two tested zirconia materials before they were chemically surface-conditioned with 1.23% APF gel. The measurement showed the following band peaks: multiple stretching vibrations for water molecules (3742–3662, 3212, 1925–1412, 564, and 472 cm^{-1}), multiple stretching vibrations for carbon dioxide (2361–2214 and 862 cm^{-1}), and multiple stretching vibrations for zirconium oxide (805 and 785 cm^{-1}).

Treating the zirconia surface with APF gel led to ester formation, confirmed by the appearance of new

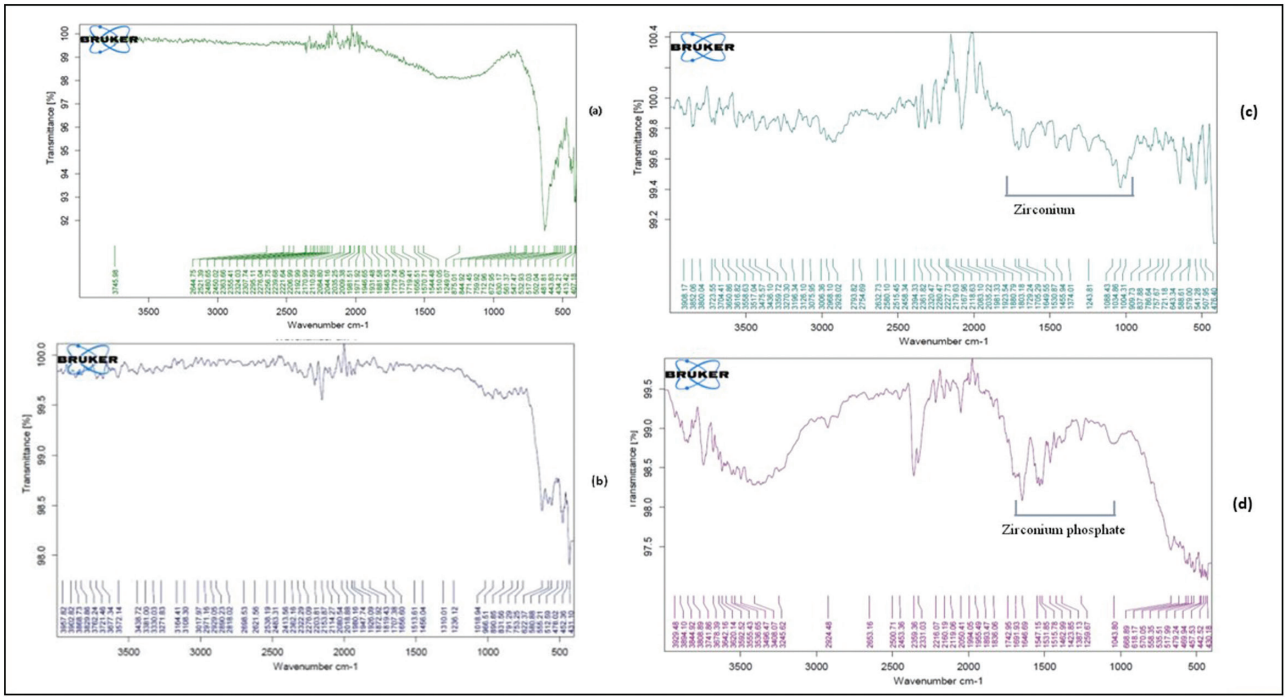


Figure 2: FTIR spectroscopy results for (a) UH and (b) BC zirconia before treatment with 1.23% acidulated phosphate fluoride gel (APF), showing multiple stretching vibrations associated with water molecules, carbon dioxide, and zirconium oxide, while (c) UH and (d) BC zirconia after treatment with 1.23% (APF) show the emergence of new absorption peaks at 1670, 1326, and 990 cm^{-1}, indicative of zirconium phosphate formation. FITR, Fourier transform infrared; UH, Upcera; BC, Burxzir

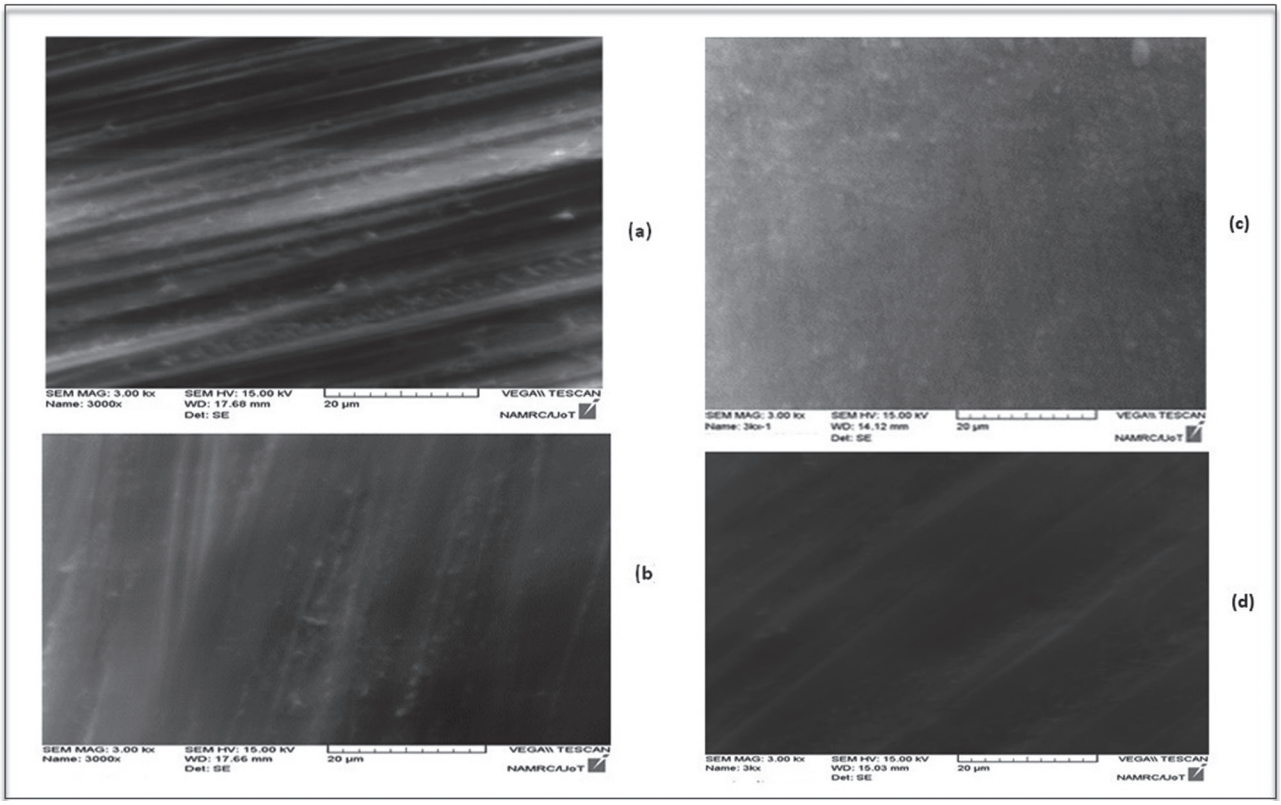


Figure 3: SEM images of the UH zirconia intaglio surface showed surface dissociation and deformation after chemical conditioning. (a) Control, (b) inner surface treated with 1.23% acidulated phosphate gel (APF), while the BC zirconia showed surface dissociation and deformation after chemical conditioning (c) control, (d) inner surface treated with 1.23% (APF). SEM, scanning electron microscope; UH, Upcera; BC, Bruxzir

Table 1: Descriptive statistics for micro-shear bond strength of Upcera and Bruxzir zirconia ceramics

Groups statistics					
Material type	Group	*N*	Mean	Standard deviation	Standard error mean
Upcera (UH)	Control	5	0.5700	0.51298	0.22941
	APF	5	1.2660	0.28615	0.12797
Bruxzir (BC)	Control	5	0.5240	0.35592	0.15917
	APF	5	1.5660	0.33374	0.14925

APF, acidulated phosphate fluoride

absorption peaks at 1670, 1326, and 990 cm^{-1}, attributed to zirconium phosphate Figure 2c and d.

2. Surface topography evaluation

The topography of the zirconia surface was examined using SEM. Figure 3a–d display images of the intaglio in the control and APF groups at a magnification of 3000X, demonstrating signs indicative of the growing deformation and dissociation in the UH and BC zirconia materials. Compared to the control group, the conditioned surfaces of the UH and BC zirconia materials displayed visible lines, scratches, and surface irregularities.

3. Micro-shear bond strength

Table 1 presents descriptive statistics, including the mean and standard deviation (SD), for different types of zirconia materials and their influence on μ-SBS of resin-modified glass ionomer cement/zirconia adhesion in both control and APF groups.

Two independent sample *t* tests ($P \leq 0.05$) were conducted to assess the significance of the results with their confidence intervals and effect size values to show the effect of sample size and mean representation of the sample groups. The mean μ-SBS values for both control and APF groups of UH and BC zirconia materials showed a significant increase after treatment with APF. Additionally, the increase was more pronounced in UH compared to BC zirconia [Tables 2 and 3].

Discussion

This study aimed to carry out a simplified clinical approach to zirconia conditioning using the APF gel for 10 min, and this time was chosen to obtain the maximum effects for the gel on zirconia, which

Table 2: Independent samples *t* tests for micro-shear bond strength test results for zirconia groups treated with APF gel

Independent samples tests					
	t	*Df*	Significance (two-tailed)	Mean difference	Standard error difference
Micro-shear bond strength test between control and APF groups for Upcera zirconia (UH groups)	−2.650	8	0.029	−0.69600	0.26269
Micro-shear bond strength test between control and APF groups for Bruxzir zirconia (BC groups)	−4.775	8	0.001	−1.04200	0.21820

APF, acidulated phosphate fluoride

enhances the luting cement and zirconia adhesion. Before making a clinical recommendation for a novel method or material, mechanical testing (i.e., μ-SBS test) should be combined with chemical (e.g., FTIR analysis) and topographical analyses (i.e., SEM analysis) of the intaglio surfaces.

Concerning Fourier transform infrared spectroscopy, in this study, the chemical and structural changes in the APF-conditioned zirconia were examined using FTIR analysis. Figure 2 depicts the outcomes of the FTIR analysis, showcasing alterations in bands, the emergence of new bands, and substantial shifts in band positions in the IR charts of the examined zirconia materials following the chemical conditioning of their surfaces. The APF gel and zirconia materials may have chemically coordinated to cause this change. Therefore, specific chemical and topographical changes emerged inside the zirconia intaglio surfaces, creating a rough etched zirconia surface appropriate for strong and durable zirconia adhesion.

The proposed mechanism suggests that when the zirconia surface was treated with the APF gel, it led to ester formation due to the ion exchange reaction with sodium phosphate (Na3PO4), resulting in zirconium phosphate.[11,18,19] Spectrum comparison before and after treatment confirmed esterification, which likely enhanced luting cement/zirconia adhesion. The proposed mechanism involves ion exchange as follows:

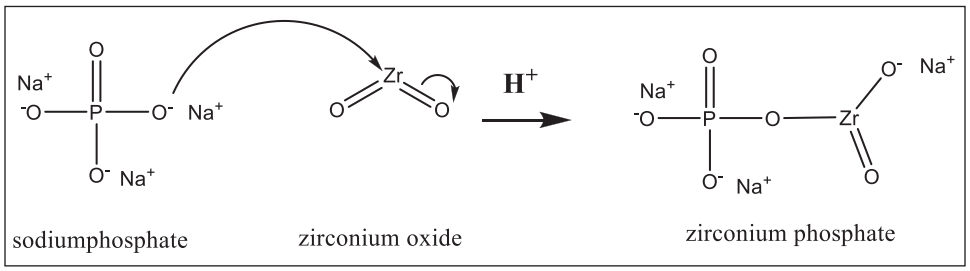
Table 3: Confidence intervals and effect size values to show the effect of sample size and mean representation of the samples groups for micro-shear bond strength test results of zirconia groups treated with APF gel

Material type	Cohen's *d* (Effect size)	Confidence interval
Micro-shear bond strength test between control and APF groups for Upcera zirconia (UH groups)	1.67	0.696 ± 0.60576
Micro-shear bond strength test between control and APF groups for Bruxzir zirconia (BC groups)	3.02	1.042 ± 0.50318

This finding is consistent with that of another study that showed the formation of a new reflection X-ray diffraction pattern due to Na3PO4 reflections after APF gel treatment of zirconia.[11]

Therefore, the first part of the null hypothesis, which posited that no chemical changes would be present on the etched zirconia surface using FTIR analysis, was rejected.

The SEM, Figure 3 examination demonstrates that the UH and BC zirconia-conditioned groups showed signs of increasing deformation and breakup. Line scratches and surface irregularities that appeared on the conditioned surfaces of the UH and BC zirconia materials may be responsible for the higher SBS than that in their control groups. This finding is consistent with those of other studies that have shown increased



SBS after the chemical condition of zirconia due to a loose porous structure, as observed by SEM.[20,21]

The SEM results of this study are consistent with those of previous studies that have shown that APF conditioning or hot acidic surface treatment causes micropores, accompanied by the appearance of scratches and alterations in the grain structure, as observed under SEM.[11,22]

Thus, the second part of the null hypothesis, which posited that the APF gel cannot alter the topography of the zirconia surface, was rejected.

Regarding micro-SBS, the conditioning of the zirconia intaglio surface with 1.23% APF gel showed a significant increase in the luting cement/zirconia bonding strength for both zirconia materials when compared to the control and APF groups [Tables 2 and 3]. Micro-irregularities found within the zirconia intaglio surface, resulting from the ion exchange of Na3PO4 between the conditioned zirconia surface and the APF gel, lead to the release of fluoride ions into the solution. This process contributes to the formation of HF acid, which subsequently induces surface roughness in the zirconia and enhances adhesion. This finding is consistent with those of other studies showing that treating zirconia with APF gel causes increased surface roughness.[11]

Another study found that the SBS of zirconia was significantly increased after chemical conditioning with HF acid, which enhances the surface roughness and increases intergrain space by eliminating peripheral atoms with low arrangement and high energy.[21] In contrast, Xie *et al.*[5,23] argued that the characteristics and surface roughness of Y-TZP are deteriorated by chemical degradation resulting from chemical conditioning rather than low-temperature deterioration. Similarly, Lee *et al.*[8] also discovered Zr–F bonds on the HF-etched zirconia.

SEM evaluation following the conditioning of the zirconia materials, as depicted in Figure 3, confirms notable changes in the intaglio surface topography, illustrating the emergence of lines, scratches, dissociation, and deformation. Moreover, cement adhesion is improved by the roughness and micro-irregularities created on the zirconia surface after chemical conditioning.[24,25]

Therefore, the third part of the null hypothesis, which posited that zirconia adhesion and μ-SBS in the context of resin-modified glass ionomer cement and zirconia adhesion would not exhibit improvement, was rejected.

Among the limitations of this study such as sample size and the cost of the samples preparation and testing and the generalizability of our findings to clinical practice, an important improvement in the adhesion of zirconia restoration to the underlying resin luting cement by using such topically applied safe gel inside the patient's mouth has also been included.

A novel, practical, cost-effective, and dependable approach for enhancing the μ-SBS of luting cement to Y-TZP ceramics is presented by analyzing the effect of APF gel on zirconia, resulting in the creation of surface micro-irregularities. This approach demonstrates altered topography and chemical composition changes in zirconia after conditioning.

Further studies are needed to evaluate the effect of using the APF gel on the mechanical and optical properties of zirconia such as tensile strength, flexural strength, cyclic fatigue, and translucency as well as other biological properties such as toxicity and compatibility.

Declaration of patient consent

Not applicable.

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Author contributions

The authors were involved in study conception, data collection, data acquisition and analysis, data interpretation, and manuscript writing, and all the authors approved the final version of the manuscript for publication.

Data availability

All data are available on request. Please address all correspondence concerning data availability to ADM at: ali.dhahi@uomosul.edu.iq.

Ethical policy and Institutional Review Board statement

This study did not involve human or animal participation. The study was performed solely on laboratory models; therefore, informed consent was not required.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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