ORIGINAL ARTICLE

The Influence of Different Types of Surface Treatment on the Surface Roughness and Bond Strength of Zirconia: An In-Vitro Study

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Abstract

Purpose: Evaluating the effects of different surface treatments on the zirconia surface and resin cement adhesive strength.

Materials and Methods: Using an STL file, 60 monolithic zirconia discs (Vita YZ HT) with dimensions of 10 mm in diameter and 2 mm in height were produced. They were machined, and sintered, and the surface was smoothed using 600, 800, and 1200 grit aluminum oxide (Al_2O_3) paper. Four groups were created based on the surface treatment applied to the discs: no treatment (control), sandblasting, potassium hydrogen difluoride, and Zircos-E solution. Resin cement cylinders (Panavia V5; Kuraray Noritake) were applied on zirconia discs using a custom mold. The shear bond strength was assessed after thermocycling. The Scanning Electron Microscope (SEM) has been utilized to analyze the morphological alterations of a specimen from every group. The results were statistically analyzed using a two-way ANOVA and a post-hoc Tukey's test ($P < 0.05$).

Results: The data analysis revealed that airborne particle abrasion with 50-µm Al_2O_3 produced the greatest shear bond strength values that were recorded at 128.933 ± 2.764Mpa. At 50.933 ± 9.573 Mpa, the control group's results were the lowest. There was a statistically significant increase in the shear bond strength values ($p<0.05$) when 50-µm Al_2O_3 was utilized in airborne particle abrasion.

Conclusion: Surface treatments increased the adhesive strength between zirconia and resin cement, and airborne particle abrasion with 50-µm Al_2O_3 was shown to be a useful technique for bond strength enhancement.

Keywords: Zirconia Surface Treatments; Potassium Hydrogen Difluoride; Shear Bond Strength; Monolithic Zirconia; Airborne Particle Abrasion.

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1. Introduction

The effectiveness of all-ceramic restorations depends on the resin material creating a strong link with the tooth structure below as well as the restoration itself. Bonding is necessary to enhance the marginal adaption, retention, bond strength, and fracture resistance of restorations [1]. Bonding additionally enhances wettability, the surface area available for bonding, and surface energy [2]. Insufficient bond strength values are formed during the manufacture or milling of the ceramic, therefore necessitating surface pretreatment [3]. The presence of micromechanical retention enhances bond strength by facilitating resin cement penetration and flow into roughened ceramic surfaces, resulting in a stronger micromechanical interlock [4]. Various tests, like shear, tensile, and micro-tensile tests, can be used to evaluate the bonding strength between dental ceramics and resin-based materials. These test procedures need to involve the addition of a load to the adhesive joints in order to create stress till the point of failure is achieved [5].

There is various treatment of the surface procedures available, including acid etching, airborne particle abrasion, grinding, diamond rotary instrument abrasion, silane coupling, silicate coating, laser, and combinations thereof [5-7]. The use of aluminum oxide (Al_2O_3) abrasive particles in airborne particle abrasion has been acknowledged as a highly efficient technique for creating a durable and enduring bond for zirconium ceramics [8]. Etching zirconium ceramic surfaces with acid has been observed to significantly change the roughness of the surface [4]. Several investigations have indicated that the use of ammonium hydrogen difluoride (NH_4HF_2) and potassium hydrogen difluoride (KHF2) for etched zirconia has been found to be successful [9].

A variety of solutions have been created to etch zirconia, one of which is a mixture of several acids that can enhance the roughness of zirconia's surface [10]. The etching solution used for zirconia is called Zircos–E Etching solution (Bio Den Co., Ltd., Seoul, South Korea). It is composed of hydrochloric acid (HCl), nitric acid (HNO_3), phosphoric acid (H_3PO_4), sulfuric acid (H_2SO_4) and hydrofluoric acid (HF) [11]. The Zircos–E Etching solution is an invention that employs ionization to produce a surface with small pores, hence improving the ability of zirconium crowns to stick to other surfaces. The etching solution improves the adhesive strength between cement and crowns made of zirconia by treating the entire surface area simultaneously [12].

The null hypothesis for this investigation posited that the choice of surface treatment has little effect on the shear bonding strength between resin cement and zirconia. This study aims to evaluate and compare the impact of different surface treatment methods, including the novel Zircos-E etching solution, on the shear bond strength between zirconia and resin cement. By examining the effectiveness of various surface pretreatment techniques, current research sought to identify the optimal approach for enhancing the durable adhesion between all-ceramic zirconia restorations and resin-based luting agents.

2. Materials and Methods

Sixty monolithic zirconia samples, namely VITA YZ® XT, Extra Translucent (VITA Zahnfabrik Bad Säckingen, Germany) were produced using presintered blocks utilizing a CAD/CAM system (Imes-Icore, GmbH, Germany). Subsequently, the samples were sintered in a specialized furnace that operates at elevated temperatures to achieve the desired final dimensions of 10 millimeters in diameter and 2 millimeters in thickness. The International Organization for Standardization (ISO) dental ceramics standards (ISO 6872, 2008) were used to establish the specimen's dimensions. The sample size was calculated with the reference of previous studies [11, 21]. It was calculated with the use of G power 3.1.9.7 (Program written by Franz- Faul, Kiel, Germany), power = 85%, alpha error of probability = 0.05 two-sided, and effect size of F was 0.48 (large effect size).

Surface Treatments: To obtain a consistent surface roughness (Ra), silicon carbide sheets with grit sizes of 320, 600, 800, and 1,200 (United Kingdom abrasive) were used to polish the bonding surfaces of the solid zirconia samples. The polishing process had been carried out utilizing a polishing machine (DAP-5, Struers, Denmark). Water cooling was used throughout the polishing process. The Ra value of every specimen was conducted utilizing a surface profilometer (Pocket Surf ®, USA). Each specimen has been measured three times across various places and orientations. The average Ra value for each sample had to be determined by taking the numerical mean of the three measurements. Ra's average value was 0.106 ± 0.040 µm. Silicon carbide sheets were used to further polish any specimen that fell outside of this range. The disc specimens were then subjected to an ultrasonic cleaning process for around three minutes in distilled water. Four groups were randomly allocated to the samples, and each group was given a different strategy for surface treatment.

The samples were categorized into four categories (n=15) based on the surface treatment performed, as described below:

1. Control Groups: There was no application of any surface treatment to the specimens.

2. Air Abrasion Group: The discs underwent air abrasion treatment utilizing particles of 50 µm Al2O3 (Cobra, Renfert GmbH, Germany) for a duration of 15 seconds. The sandblasting machine (KXC-IIB, China) was used for the process, with a 4 bar pressure and a distance of 10 mm.

3. KHF2 Groups: KHF2 powder was applied to the zirconia surfaces in an amount of around 70.0 (±15.0) milligrams. The samples containing KHF2 were subjected to heating inside a porcelain furnace (VITA ZYRCOMAT© 6000 MS, Bad Säckingen, Germany) at a temperature of 280ºC. The surface for bonding was subsequently cleansed utilizing a steam cleaner for a duration of 15 seconds, followed by a period of 15 seconds of compressed air.

4. Etching Group: The samples were submerged in the Zircos–E Etching solution for a duration of 2 hours as per the instructions provided by the manufacturer. Subsequently, they were washed with cold tap water.

Following the surface had undergone treatments, zirconia discs underwent a cleaning process using an ultrasonic cleaner (Sunshine, GuangZhou, China) with distilled water for a duration of 10 minutes.

Bonding procedure: For this study, an acrylic block was constructed by using a custom-made square elastomer mold measuring 1 cm x 1 cm x 2 cm. The untreated surface of the specimen was immersed in a mixture of cold cure acrylic (Duracryl® Plus, Spofa Dental, Czech Republic) leaving approximately 2 mm of the treated surface of the zirconia disk exposed. Prior to cementation, a thin layer of Clearfil Ceramic Primer Plus (Kuraray, Osaka, Japan) was added through a disposable, clean brush each time on the surface of all the samples for 1 minute, then blown dry with air. Custom-made Teflon molds were used. It had a diameter measuring 2.5 mm and an overall height of 3.5 mm. The resin cement (Panavia V5; Kuraray Noritake) will be automatically mixed using a mixing tip and then applied into the mold until it completely fills up and is covered with a flat glass (Figure 1). Ultimately, the light polymerization process was carried out for a duration of 20 seconds, following the manufacturer's recommendation, utilizing the Eighteeth (Changzhou Sifary Medical Technology Co., Ltd, China). The polymerization was conducted from four distinct directions. The Teflon mold was removed after polymerization and the prepared samples, with resin cement cylinders attached (Figure 2).

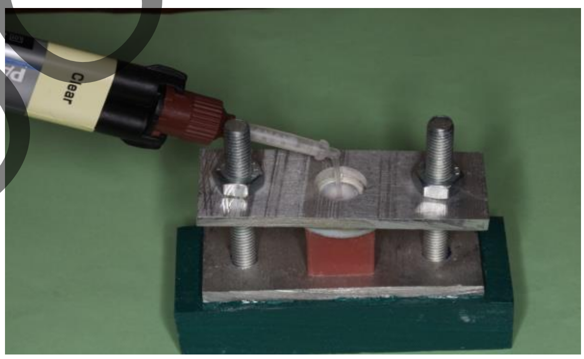

Figure 1. Injection of Panavia V5 resin cement E

Figure 2. Final zirconia-resin cement samples

2.1. Thermal Aging Technique

The samples have been immersed in distilled water and kept at an ambient temperature for a duration of 24 hours. The samples were heated to a temperature that ranged from 5 to 55 °C for 5000 cycles in water that had been deionized. There were dwell and transfer intervals of 20 seconds in every cycle. (with the SD Mechatronic GmbH, Westerham, Germany, SD Mechatronic Thermocycler) [13].

2.2. Shear Bond Strength

The SBS tests have been carried out using universal testing equipment (Tinius Olsen, H50KT, UK). The adhesive contact was subjected to shear stress at a crosshead speed of 0.5 mm/min up till debonding. To calculate the shear bond strength in Megapascals (MPa), the failure load (N) was divided by the resin cement area (πr2) [3,14].

2.3. Stereomicroscope Examination

The broken surfaces of zirconia specimens have been examined using a stereomicroscope (Zumax OMS2380, Zumax Medical Co Ltd, China) at a magnification of ×15. Depending on where the failure happened, the failure mechanisms have been divided into cohesive, adhesive, or mixed categories. Adhesive failure was discovered at the boundary between the cement and zirconia, cohesive failure occurred in the resin cement and mixed failure involved both cohesive and adhesive components. When there was a combination failure, the residual resin cement partially covered the zirconia surface [15].

2.4. Scanning Electron Microscope Analysis

In addition to the 60 samples used in the study, an extra sample from each treatment group, including one without treatment, underwent analysis with a scanning electron microscope (Inspect F50, FEI, USA) to assess their similarities in dimensions. Prior to an assessment, the top surface specimens were coated using a layer of gold by the sputter-coating process. The assessment was conducted at several magnifications, ranging from ×10000 to ×150 (11).

3. Results

Table 1 provides an overview of each group's standard deviations and mean shear bond strength data. The results of the 2-way ANOVA show that the bond strength values were significantly impacted by the surface treatment ($P < 0.05$), as shown in Table 2.

With each surface treatment, the adhesive strength between resin cement and zirconia increased. Based on statistical analysis, the sandblasting group's monolithic zirconia block bonding strength was shown to be substantially higher than that of the other groups. However, Table 3 shows that there was no appreciable alteration in the bond strengths between the KHF 2 group and the etching group.

There is a strong correlation between the distribution of the mode of failure and various categories. The KHF2 group had the highest adhesive, followed by the control group, while the sandblasting group had the lowest. The sandblasting group had slightly higher cohesion than the control group and no cohesion in the KHF2 groups, while the etching group

Table 1. Shear bond strength (MPa) descriptive statistics

Groups	Minimum	Maximum	Mean	±SD	±SE
Group A No treatment (control group)	40.000	65.000	50.933	9.573	2.472
Group B Air-borne particle abrasion with 50-µm Al_2O_3.	125.000	133.000	128.933	2.764	.714
Group C potassium hydrogen difluoride KHF_2	75.000	90.000	82.533	4.121	1.064
Group D Zircos- E 120 min	70.000	90.000	78.667	6.935	1.791

Table 2. One-way-ANOVA between groups (A, B, C & D)

Groups	Sum of Squares	df	Mean Square	F	P value
Between Groups	47048.800	3	15682.933	381.690	**0.000**
Within Groups	2300.933	56	41.088	-	-
Total	49349.733	59	-	-	-

Table 3. Tukey HSD test among different groups

Groups		Mean Difference	Lower Bound	Upper Bound	P value
A	**B**	-78.000	-84.198	**0.000***	-71.802
	C	-31.600	-37.798	**0.000***	-25.402
	D	-27.733	-33.931	**0.000***	-21.536
B	**C**	46.400	40.202	**0.000***	52.598
	D	50.267	44.069	**0.000***	56.464
C	**D**	3.867	-2.331	0.359^	10.064

* indicates Significant at $p<0.05$, and ^ indicates not significant at $p>0.05$.

Table 4. Modes of failure of the different groups

			Groups				Fisher exact	P value
			A	B	C	D		
Mode of failure	**Adhesive**	N.	7	0	9	2	16.007	**0.009 Sig.**
		%	46.67	.00	60.00	13.33		
	Cohesive	N.	3	5	0	3		
		%	20.00	33.33	0.00	20.00		
	Mixed	N.	5	10	6	10		
		%	33.33	66.67	40.00	66.67		

had no cohesive. Sandblasting group and etching group with higher mixed followed by KHF2 groups with lower in the control group (Table 4).

Figure 3 displays SEM pictures of the zirconia groups following surface treatment.

Specimens in the control group have a surface that is generally smooth (Figure 3a). As shown in Figure 3b, each sample showed a topographic morphology after being abraded by airborne particles. These topographic morphologies were defined by evenly degraded, edge-shaped micro-rough surface textures that contained shallow fissures with scattered micro-irregularities. The KHF2 group displayed a surface morphology characterized by surface irregularities, micro-porosities, and micro-stretches, as shown in Figure 3c. The etching group samples displayed images that were distinct from those of different groups. The samples within that group exhibited the development of micropores with varying widths and depths, as illustrated in Figure 3d.

4. Discussion

Shear bond strength was significantly increased when 50 µm Al_2O_3 particles were applied to the adhesive side of zirconia in comparison to the other groups. This behavior may be explained by the fact that sandblasting the surface of the zirconia adhesive increases surface irregularities and creates depressions. The results of the present study are consistent with those of Cavalcanti *et al.* [16], which exhibited an increase in the intensity of bond strength following the application of air abrasion using 50 µm Al_2O_3. However, the research conducted by de Oyague *et al.* [17] contradicts the notion that using air abrasion over the surface that bonds of a zirconia substrate results in higher bond strength, even though it does roughen the surface of the substrate in comparison to the control group. The difference observed could be ascribed to differences in grain size or the magnitude of pressure exerted during the investigation.

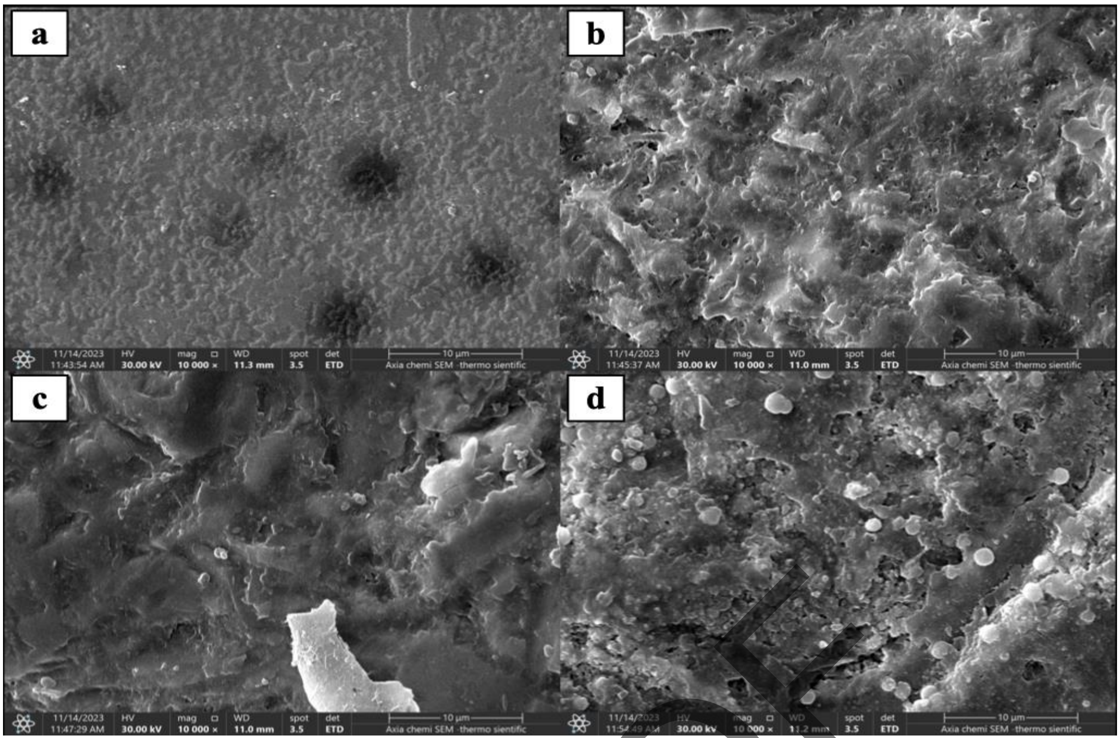

Figure 3. SEM at 10000x magnification a) Group A specimen (Un-treated zirconia), b) Group B zirconia specimen (airborne-particle abraded surface), c) Group C zirconia specimen (potassium hydrogen difluoride KHF_2), d) Group D zirconia specimen (Zircos-E treatment)

This study's findings indicate that the use of potassium hydrogen difluoride on the bonding surface of zirconia greatly increases shear bond strengths in comparison with the Zircos-E solution group and the control group. The enhancement in performance is probably attributed to the surface roughness along with imperfections within the zirconia bonding surface, which promote a stronger interlocking with the resin cement.

This result is consistent with the findings of Ruyter *et al.* [9], who found that a rough etched surface that promotes strong and long-lasting adhesion was produced by melting NH4HF2 and KHF2 over ground and polished Y-TZP. Furthermore, this result is consistent with the study carried out by Akazawa *et al.* [18], who found that the surface roughness of zirconia causes a significant increase in shear bond strength when KHF2 melts on its bonding surface. However, the investigation's findings showed that sandblast treatment was associated with lower bond strength values.

Based on the statistical analysis conducted in this experiment, the average shear bond strength for the Zircos E etching solution group was significantly lower than that of the other groups. In a study by Sadid-Zadeh *et al.* [11], it was shown that using the Zircos E etching solution reduced the mean shear bond strengths of resin cement to zirconia in comparison to air particle abrasion. In research by Cho JH, [10] the shear bond strength of zirconia was compared to different resin cements; air abrasion, tribochemical silica coating, and the Zircos E etching solution were applied to the zirconia surfaces. Additionally, their results showed that samples treated with air abrasion had an average shear bond strength greater than samples treated with the Zircos E etching solution due to the use of Panavia as a luting medium. This paper shows that Panavia F 2.0's chemical bonding mechanism accounts for its much lower bond strength in the Zircos E etching system group. The hydroxyl group that is present across the zirconia surface interacts with the MDP component of Panavia F 2.0. Previous studies have shown that under heat cycling circumstances, this response is not efficiently maintained [19, 20]. It is possible to suggest that thermocycling zirconia eliminated the hydroxyl group from its surface, resulting in a reduction in Panavia F 2.0's shear bond strength.

Sales *et al.* [21] examined how the surface characteristics and micro-shear bond strength of transparent and opaque zirconia were affected by air abrasion, etching with the Zircos E etching solution, and a combination of these two surface treatment techniques. Their results showed that, in comparison to the air abrasion method alone, the application of etchant alone or in conjunction with air abrasion produced a much higher shear bond strength value for zirconia. The reason for the contradictory findings in this research might be because RelyX Ultimate, a luting agent from 3M ESPE in St. Paul, Minnesota, USA, was used instead of Panavia V5, the luting agent used in this study. Furthermore, it is essential to recognize that the study was limited by the use of just two samples per group.

When looking at the different forms of failure that occurred during the shear test, it was found that the control and KHF2 groups mostly showed adhesive failure, whereas the sandblasting and the Zircos E etching groups tended to show mixed failure. The bond strength values were also supported by the failure mechanisms. It was shown that mostly adhesive failures occurred in the groups with lower bonding strength values and primarily mixed failures occurred in the groups with higher bond strength values.

5. Conclusion

Within the limitations of this experiment, it is clear that the surface treatments strengthened the connection. In addition, the specific kind of surface treatment had a major effect on the shear bond strength between zirconia and resin cement. The adhesive strength between zirconia and resin cement may be greatly enhanced by using air-abrasion with 50 µm Al_2O_3, as opposed to other surface preparation techniques.

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