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The Influence of Surface Treatment with Zircos–E etching solution and Sandblast on The Surface Roughness of Zirconia (An in Vitro Study)

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Abstract

Purpose: To evaluate the effect of Zircos–E etching solution and sandblasting with Al2O 3particle on the roughness of zirconia ceramic**.**
Material and methods: 45 monolithic zirconia discs (Vita YZ HT) with measurements of 2 millimeters in height and 10 millimeters in diameter were produced. All samples were machined, sintered, and the surface of each disc was smoothed using 600, 800, and 1200 grit aluminum oxide paper. Three groups were created based on the surface treatment applied to the discs, group (A): no treatment (control), group (B): sandblasting, and group (C) Zircos-E solution. Using a profilometer, the zirconia's surface roughness has been investigated for each group. The scanning electron microscope (SEM) has been utilized to analyse the morphological alterations of a specimen from every group. One-way ANOVA and Tukey HSD tests have been utilized to assess the data at the 5% significance level.
Results: The zirconia surface roughness value was significantly increased by sandblasting with 50µm AL2O3 (1.921 µm). The zirconia surface roughness value did not significantly change after chemical treatment with Zircos-E etching solution (0.910 µm), as compared to the control group (0.601 µm).
Conclusion: Acid etching has little impact on zirconia surface roughness, whereas zirconia surface roughness increases after AL2O3 sandblasting.

Keywords: Zirconia surface treatments, Surface roughness, Zircos–E etching solution, Monolithic zirconia, Airborne particle abrasion.

Introduction

Zirconia ceramic's exceptional structural qualities, including its aesthetic, biocompatibility, and high mechanical capabilities, have made it a successful material for dental prostheses (Ghasemi et al., 2014). Although implant abutment should be appropriately attached to tooth tissues, zirconia was reportedly difficult to bond with these areas in contrast to silica-based ceramic materials (Aboushelib et al., 2010). In addition, the polycrystalline nature of zirconia is quite stable; however, one drawback is that it doesn't lend itself to much surface treatment as compared to glassy materials (Melo et al., 2015).

Multiple studies are reporting poor adherence to cement on low-surface-roughness zirconia (Mattiello, Vivaldi, et al., 2013). Mean roughness values of untreated zirconia differ within the range from 0.2 to 0.98µm, and therefore further efforts/techniques trying to increase surface roughness are still in development (Candido et al., 2014). The adhesion between luting resin and the restoration will be increased when the surface of cement is roughened because this makes it more receptive to adhesive, a finding supported by several investigations (El-Shrkawy et al., 2013). Several strategies and materials have been suggested in the literature to improve its adhesion to resin cement (Stefani et al., 2015).

There are already standardized methods of modifying the surface of zirconia to get a reliable bonding, such as adapting it mechanically and chemically (Martins et al., 2019). The most common methods and materials include lasers, acid etching solutions, tribochemical silica coating (TSC), air abrasion with alumina particles, surface grinding using diamond rotary tools, and selective infiltration etching (SIE). It has been stated that the procedure of sandblasting using 50-125 µm AL2O3 particles is essential in ensuring proper adhesion between zirconia and resin cement (El-Shrkawy et al., 2013). Some investigators have claimed that air abrasion treatment with aluminum oxide (Al_2O_3) particles before luting zirconia is necessary even if unconventional primers and universal adhesives were used (Zens et al., 2019). However, the application of sandblasting introduces defects and alters the surfaces resulting in inducing a phase transition leading to changes at the surface level this may affect zirconia mechanical properties (Usumez et al., 2013).

To reinforce the zirconia bond, the Zircos-E etching solution (BIO DEN Co, Ltd, Geumcheon-gu, Seoul, South Korea) was introduced. It is a solution that is composed of many acids, including hydrofluoric acid, hydrochloric acid, sulfuric acid, nitric acid, and phosphoric acid. It was able to roughen and modify the topography of the zirconia surface (Sadid-Zadeh et al., 2021; Cho et al., 2017). Another study by Cho et al. (2017) also showed the surface roughness of zirconia after 2 h in an expanded Zircos etching process. Xie et al. (2015) similarly conducted a very interesting study investigating the impact of acidic solutions on mechanical properties and the results were also promising. Consequently, acid etching may represent a superior approach to sandblasting as an efficient means of fabricating surface imperfections in zirconia ceramic. Therefore, improving surface roughness parameters in prosthetic materials such as zirconia seems essential for a reliable cement interlock.

Surface roughness evaluation can be performed on both macro-scale and micro levels. Therefore, different non-contact devices like an atomic absorption microscope, SEM, or confocal laser microscopy could be used for this aim (Bagby & Jones, 2013). The profilometer, as one of the oldest and most commonly used contact-imaging techniques with inexpensive cost and feasibility for study is generally applied to assess surface irregularities in zirconia after treatments (Kirmali et al., 2014).

In other words, if we expose a surface to sandblasting, may have macroscopical alterations, and by acid etching, microscopically changes can be generated. Thesis to be tested: The surface roughness of zirconia will not change following acid etching or sandblasting. Aim of the study: The objective of this in vitro experimental dental laboratory research was to evaluate and compare surface smoothness values on sintered zirconia ceramic surfaces following etching with Zircos-E or sandblasting using aluminum oxide particles.

The null hypothesis tested in this study is that there are no differences in roughness between different treatment techniques

Material And Methods

A CAD/CAM system (Imes-Icore, GmbH, Germany) was used to generate 45 monolithic zirconia samples, namely VITA YZ® XT, Extra Translucent (VITA Zahnfabrik Bad Säckingen, Germany), using presintered blocks. The samples were then sintered in a special furnace that runs at high temperatures to produce the final dimensions that are needed, which are 2 millimeters in height and 10 millimeters in diameter. The specimen's measurements were determined using the International Organization for Standardization (ISO) dental ceramics standards (ISO 6872, 2008). Three groups were randomly allocated to the samples, and each group was given a different strategy for surface treatment.

Zirconia samples had been categorized into 3 categories (n=15) depending on the surface treatment performed, as described below:

1. **Control Groups**: There was no application of any surface treatment to the samples.
2. **Air Abrasion Group:** The discs underwent air abrasion treatment with particles of 50 µm aluminum oxide (Cobra, Renfert GmbH, Germany) for fifteen seconds. The sandblasting machine (KXC-IIB, China) was used for the process, with a 3 bar pressure and ten millimeters apart.
3. **Etching Group:** The samples were submerged into 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.

Then, zirconia discs underwent a cleaning process using an ultrasonic cleaner (Sunshine, GuangZhou, China) with distilled water for 10 minutes. Sample size calculation was done by Using G power 3.1.9.7 (Program written by Franz-Faul, University of Kiel, Germany) With power of study=85%, alpha error of probability=0.05 two-sided, effect size of F is 0.4 (Large effect size), three groups with all these conditions the sample size is about 45 samples (15 for each group). Effect size F are: Small =0.1, medium=0.25, large=0.4. (Cohen, 2013).

Surface roughness measurement

For every specimen, a mean roughness profile (Ra) was analyzed to characterize the overall surface roughness. Using a contact stylus profilometer, the surface roughness of each specimen was determined quantitatively in micrometers (µm), as seen in Figure 1. Each disc specimen's center was measured three times in a row parallel to one another, and the mean to ascertain the specimens' general surface characteristics, Ra was calculated (Incesu and Yanikoglu, 2020).

The profilometer's roughness assessment parameters were a display resolution of 0.01 µm, a traverse speed of 5.08 mm per second, a maximum stylus force of 1500 mgf /15.0 mn, a traverse length of 5.0 mm, a cut-off value of 0.8 mm, and measurement accuracy meet ASME B46 .1, ISO standards (Flury et al., 2012). Data description, analysis, and presentation were performed using Statistical Package for Social Science (SPSS version -22, Chicago, Illinois, USA), minimum, maximum, mean, standard deviation, Standard error, Shapiro Wilk test for normality, One Way ANOVA with Tukey HSD posthoc test, level of significance is when p-value less than 0.05.

Scanning electron microscope (SEM) analysis

Apart from the 45 samples that were 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. The sputter-coating method applied a layer of gold to the top surface specimens before evaluation. The evaluation was carried out at many magnifications, ranging from ×10000 to ×150 (Sadiq-Zadeh et al., 2021).

Results

The standard deviation (SD) and mean for zirconia (Ra) surface roughness values after various surface treatment methods are shown in Table 1. Surface roughness is higher in air abrasion groups (1.922) followed by the etching group (0.910) while lower in the control group (0.600) with significant differences among groups. Using multiple pairwise comparisons (Tukey HSD), all results appeared to show that there were statistically significant results between groups (Table 2).

Figure 2 displays scanning electron microscope (SEM) pictures of the zirconia groups following surface treatment. Specimens in the control group, which have a surface that is generally smooth, were found to have markings and rough places after they were cut and ground (Figure 2a). As shown in Figure 2b, 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 etching group displayed a surface morphology characterized by surface irregularities, microporosities, and microstretches were observed, as shown in Figure 2c.

Discussion

A crucial element in the clinical effectiveness of fixed prostheses is the long-lasting connection between zirconia and resin cement (Bottino et al., 2014). Reliable surface roughness is necessary to attain micromechanical retention between cement and zirconia ceramic to accomplish this aim (Melo et al., 2015; Tzanakakis et al., 2016). This has prompted investigators to examine how different surface treatments affect surface roughness (Kirmali et al., 2014). The non-contact method's inability to measure bright surfaces accurately at times stems from the reflected light's dispersion effect, which can lead to reading errors (Lee et al., 2019). As a result, a contact method with a profilometer was utilized in the present study. The improvement in surface roughness indicates a larger surface area, which is essential for increasing the connection between the resin cement and the indirect restoration (Petrauskas et al., 2018). This investigation discovered that the treatments used significantly impacted the zirconia ceramic's roughness. The investigated treatment procedures demonstrated statistically significant variations in Ra values, rejecting the null hypothesis of this research, which maintained that there are no changes in roughness across various treatment strategies. The data show that sandblasting with aluminum oxide particles with a cross-section of 50–125 µm is the main zirconia surface treatment performed before the cementation process (Melo et al., 2015). Since many previous investigations have established that four bars of pressure with 50 µm of AL2O3 particles are an adequate number to produce enough binding strength without causing unnecessary damage, we utilized this amount for the present study (Grasel et al., 2018). In the past, airborne particle abrasion was used to create a rougher surface while also cleaning the surface and removing impurities. Additionally, this process will modify the surface, increasing the luting material's wettability and surface energy (Usumez et al., 2013). By making these adjustments, the resin cement will be able to penetrate these micro-retentions and strengthen the micromechanical interlock (Xie et al., 2015). Roughness and retention were correlated, meaning that a rise in roughness also results in a rise in retention (Kirmali et al., 2014; Su et al., 2015). This study corroborated with the findings of a few other studies which found that alumina oxidative particle blasted zirconia was a much better surface treatment process to enhance roughness morphology on Zr remained the same (Kirmali et al., 2014; Su et al. Nevertheless, air abrasion can bring some damage to the zirconium dioxide structure, as pointed out by previous studies, and an alternative technique, which is acid etch was also investigated due to this concern (Subaşı & İnan). A surface treated through acid etching is a technique where particles are dissolved on the zirconia surface chemically due to the use of stock acid. This is typically more favorable than sandblasting as it allows for a less subjective application and results in more uniform outcomes (Kukiattrakoon et al., 2011). Zircos-E etching solution, an evaluation method to create surface alterations on the non-glass ceramic prosthesis by the process of office, showed in contact with zirconia at 20 min. An earlier study showed changes in the surface of zirconia after being subjected to a similar technique, stating higher bond strength and thereby increasing the possibility of micro-mechanical retention with luting resin cement (Hasan et al., 2019). The data revealed that the finish line for both the etching and control groups showed no significant difference in mean Ra value. This suggests that zirconia was not roughly treated by the Zircos-E etching solution in profilometric evaluation. Thus, probably the results of this study will be reliable compared to the other studies where hydrofluoric acid etching is used in concentrations from 4–10%. This research revealed that an acid solution with a low concentration for short-term preparation in zirconia did not have any effects on the surface morphologies and roughness that were highly significant (Sriamporn et al., 2014). Nevertheless, Sriamporn et al. (2014) demonstrate that in low concentrations, raising the application time and concentration temperature of hydrofluoric acid may create a higher surface roughness. It will be quite beneficial to look for a secure and efficient method of producing surface roughness, such as using a stronger acid than hydrofluoric acid. Despite these results, other studies concluded that without strengthening the bond, acid etching generated nano-irregular pattern zirconia. The rationale for their reasoning was that the etched zirconia surface's nano-porosities prevented the high viscosity of the resin cement from penetrating them (Smielak and Klimek, 2015). According to the study's findings, sandblasting increases surface roughness within the profilometric range, whereas twenty minutes of Zircos–E etching solution did not.

Conclusion

Within the limits of the present study, using AL2O3 (50µm) sandblasting, mechanical zirconia surface treatment, generated rougher zirconia surfaces compared to acid etching with Zircos–E etching solution.

Conflict of interest

The authors reported that they have no conflicts of interest.

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Figure 1. Profilometer measuring the surface roughness of a specimen.

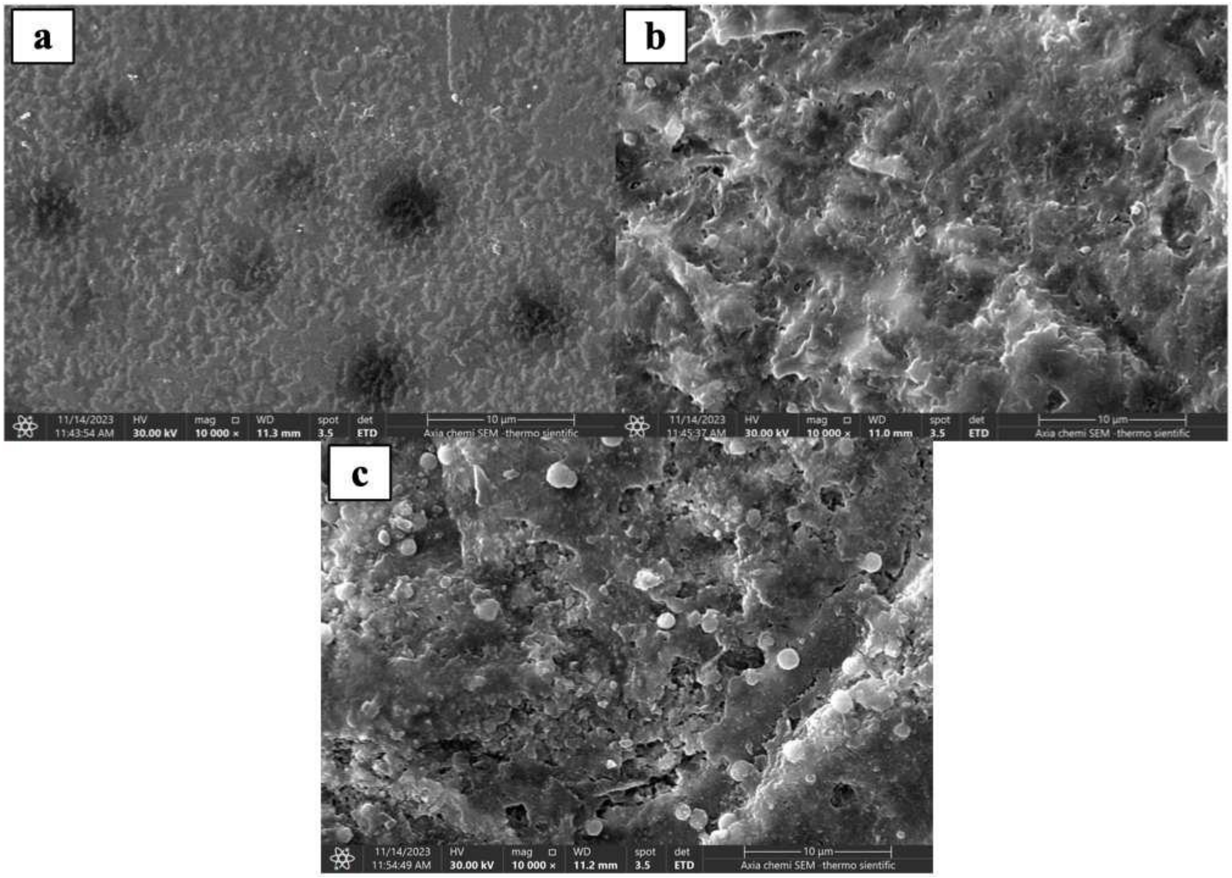

Figure 2. SEM at 10000x magnification a) Group A specimen (Un-treated zirconia), b) Group B zirconia specimen (airborne-particle abraded surface), c**)** Group D zirconia specimen (Zircos-E treatment).

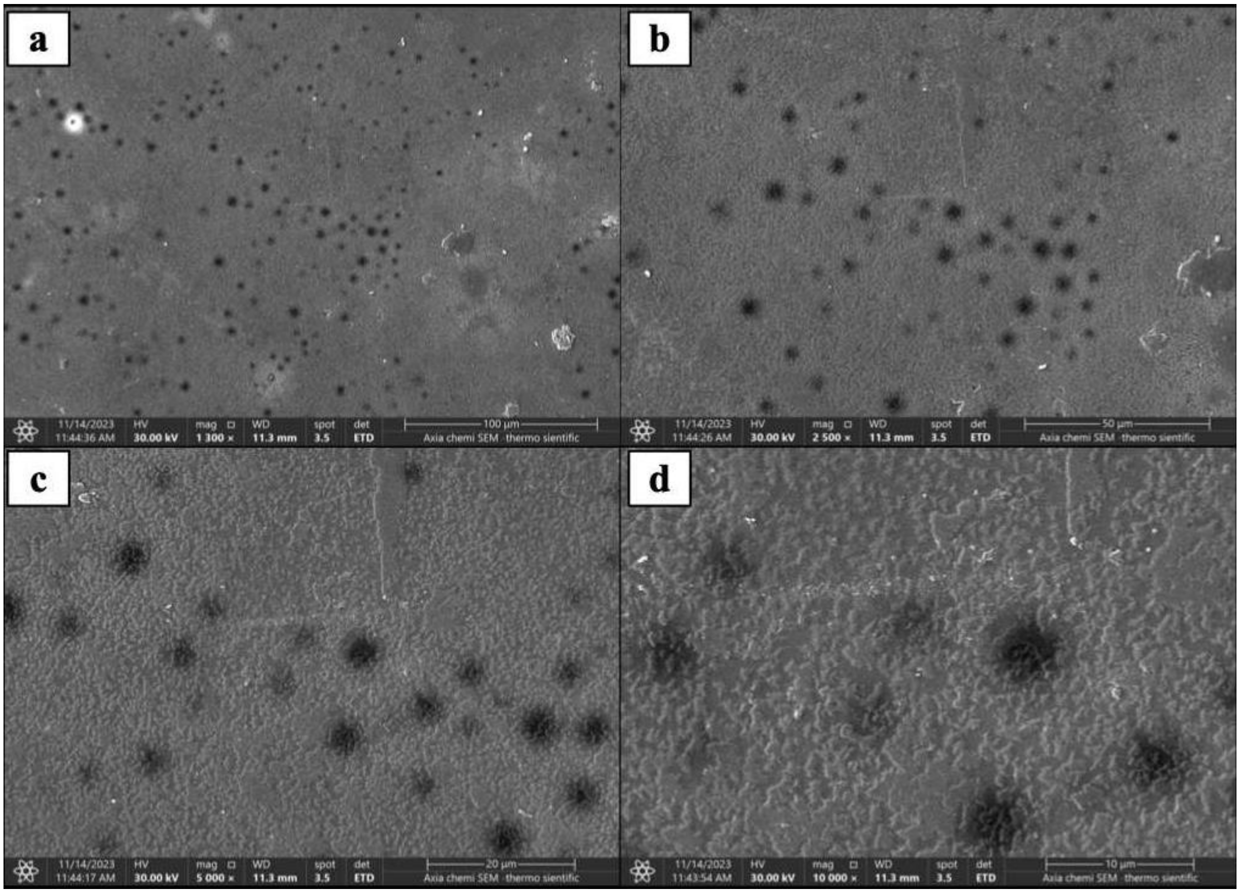

Figure 3. SEM for Group A zirconia specimen (Un-treated zirconia) at a) 1300x magnification, b) 2500x magnification, c) 5000x magnification, d) 10000x magnification.

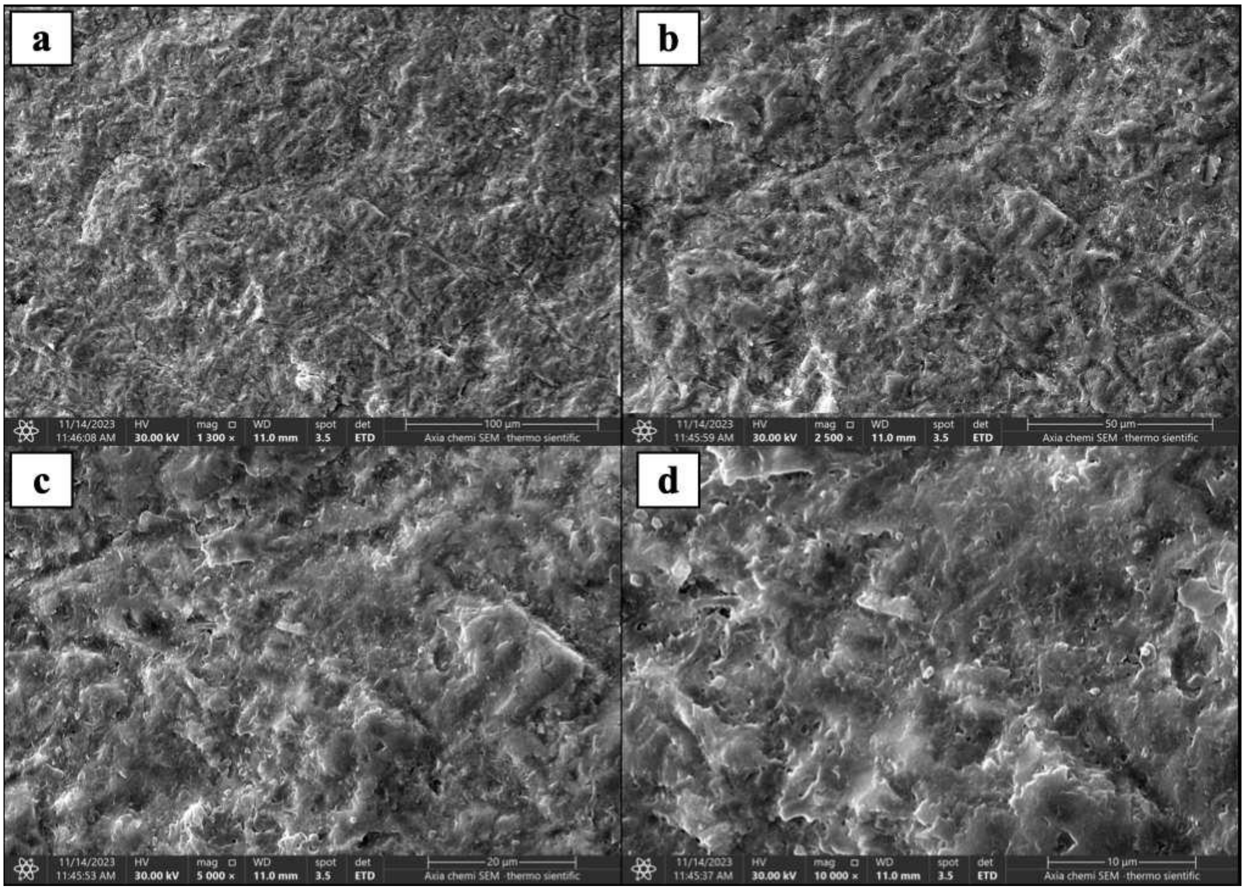

Figure 4. SEM for Group B zirconia specimen (airborne-particle abraded surface) at a) 1300x magnification, b) 2500x magnification, c) 5000x magnification, d) 10000x magnification.

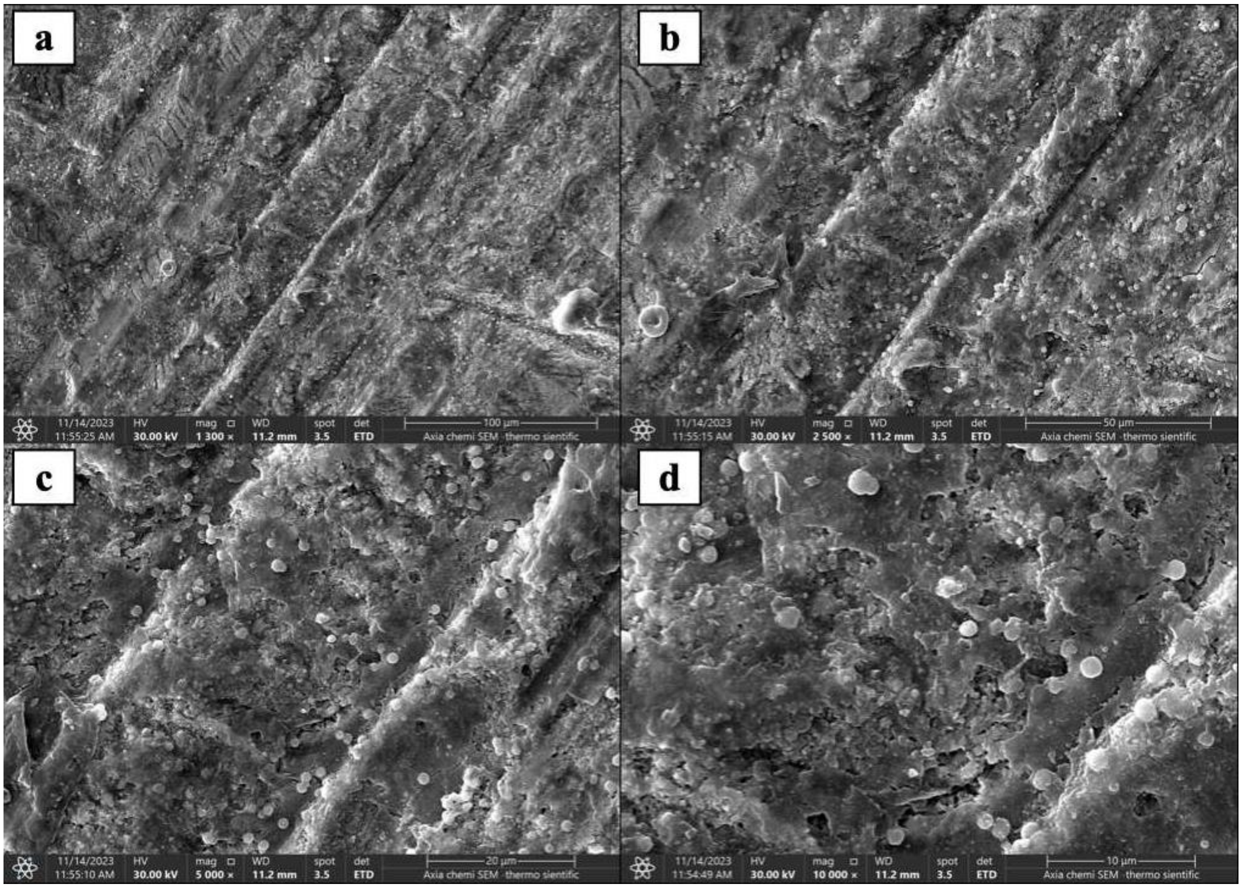

Figure 5. SEM for Group C zirconia specimen (Zircos-E treatment) at a) 1300x magnification, b) 2500x magnification, c) 5000x magnification, d) 10000x magnification.

Table 1. Descriptive statistic of the surface roughness in micrometers (µm) for three different surface treatments

Descriptive and statistical test of surface roughness (Ra) among groups using One Way ANOVA.

Groups	N	Mean (µm)	±SD	SE	Minimum	Maximum	F	P value
Control	15	0.600	0.105	0.027	0.417	0.803	98.752	0.000 Sig.
Air Abrasion	15	1.922	0.335	0.086	1.397	2.427		
Etching	15	0.910	0.307	0.079	0.443	1.283		

Sig., significant at $p<0.05$.

Table 2. Multiple pair Comparisons of surface roughness (Ra) among groups using Tukey HSD

(I) Groups	(J) Groups	Mean Difference (I-J)	P value*	95% Confidence Interval	
				Lower Bound	Upper Bound
A	B	-1.32133	**0.000**	-1.5602	-1.0825
	C	-.31000	**0.005**	-0.5307	-0.0893
B	C	1.01133	**0.000**	0.7141	1.3086

*= significant at $p<0.05$.