

COMPARATIVE EVALUATION OF SURFACE TREATMENT METHODS ON THE SURFACE ROUGHNESS OF MONOLITHIC AND MULTILAYERED ZIRCONIA-AN IN VITRO STUDY

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ABSTRACT

Objective: To comparatively evaluate the effect of different surface treatment methods on the surface roughness of monolithic zirconia and multilayered zirconia in an in vitro study. **Materials and methods:** A total of 36 zirconia samples were fabricated and divided into six groups, with each group consisting of six disk-shaped samples (16mm x 2mm thickness). The groups included are untreated monolithic zirconia, sandblasted monolithic zirconia, acid etch monolithic zirconia, untreated multilayered zirconia, acid etch multilayered zirconia and sandblasted multilayered zirconia. Surface roughness assessed using profilometer to evaluate surface changes post. **Results:** Untreated monolithic and multilayered zirconia showed the lowest roughness values. Sandblasting increased surface roughness in both zirconia types by creating micro-irregularities on the surface. The sandblasted monolithic zirconia group exhibited the highest roughness values ($2.29 \pm 0.62 \mu\text{m}$), followed by sandblasted multilayered zirconia ($0.62 \pm 0.18 \mu\text{m}$). **Conclusion:** Surface treatment significantly influences the surface characteristics of zirconia restorations. Sandblasting was more effective than acid etching in increasing surface roughness of both monolithic and multilayered zirconia. The increased roughness enhances micromechanical retention and may improve the bond strength of resin cements. Therefore, sandblasting can be considered a preferred surface treatment for achieving durable zirconia restorations.

Keywords: Surface roughness, Zirconia, Profilometer, Multilayer

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INTRODUCTION

In the ever-evolving field of dentistry, technological advancements have significantly transformed the design and fabrication of dental restoration. One such breakthrough is the integration of CAD/CAM technology, which has greatly improved the precision, consistency, and efficiency of prosthesis fabrication. The evolution of computerized systems, combined with the development of novel ceramic microstructures, has brought a major shift in the clinical workflow for both dentists and technicians, while also expanding treatment options for patients[1]. A notable advancement has been the introduction of monolithic restorations produced from high-strength ceramics such as zirconia. Although metal-ceramic restorations continue to be regarded as the gold standard for tooth restoration, the growing demand for superior aesthetics has led to an increased preference for metal-free alternatives[2]. Zirconia often referred to as "ceramic steel"[3]. It is a crystalline form of zirconium dioxide that offers metal-like mechanical properties along with tooth like color and translucency[4]. It has been used in dentistry for orthodontic applications, crowns, bridges, and implant frameworks. 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 [5]. There is various treatments of the

surface procedures available such as sandblasting, acid etching, grinding, diamond rotary instrument abrasion, silane coupling, silicate coating, laser, and combinations [6,7]. Air-borne particle abrasion treatment uses the kinetic energy of abrasive grains in a stream of compressed gas (usually air). The accelerated abrasive grains (Al_2O_3 grains) strike the surface of the treated surface (zirconium dioxide) leading to micro-cutting and surface preparation through material removal. This process is termed abrasion, and the treatment is referred to as abrasive blasting[8]. 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[5]. Hydrofluoric acid (HF) treatment is commonly used on silica-based ceramics to react with, and remove, the glassy matrix that contains silica. This leaves the crystalline phase exposed, generating surface roughness. This process also results in enhanced surface energy on the ceramic surface, which allows greater penetration of the resin cement tags, increasing the bond strength between the cement and ceramic[9]. Etching zirconium ceramic surfaces with acid has

been observed to significantly change the roughness of the surface. Zirconia-based restorations are available mainly in two forms: porcelain-veneered zirconia (bi-layered zirconia) and fully contoured zirconia (monolithic zirconia). More recently, multilayered zirconia has been introduced in the form of blanks and blocks intended for full-contour crowns, single units, and multi-unit bridges. While multilayered zirconia offers enhanced esthetics compared with monolithic zirconia, its mechanical properties remain a subject of ongoing evaluation, especially given its relatively recent entry into the market. The objective of this in vitro study is to comparatively evaluate the influence of different surface treatment methods on the surface roughness of monolithic and multilayered zirconia. The null hypothesis states that there is no statistically significant difference in surface roughness between monolithic and multilayered zirconia, irrespective of the surface treatment method applied.

MATERIAL AND METHODOLOGY

The study was designed in compliance with the institutional ethical committee's codes of research ethics and obtained the approval with the reference number IEC-PDCH 8/7-2025. The present in vitro study was conducted at the White Lab – Material Research Centre, Saveetha Institute of Medical and Technical Sciences (SIMATS). A power analysis was performed using G*Power software version 3.1.9.4 to determine the minimum sample size required for the study, based on data obtained from a previously published reference article. Considering a study power of 95%, an alpha error probability (α) of , the analysis indicated that a minimum of six specimens per subgroup was required. Accordingly, zirconia samples were divided into six groups, with each group comprising six disk-shaped specimens measuring 16 mm in diameter and 2 mm in thickness, resulting in a total of 36 samples ($n = 6$ per group). The grouping of samples

was performed according to the different surface treatment protocols evaluated in the study. Samples were fabricated from presintered zirconia blanks (CERAMOTION) were designed with the help of CAD software (Exclusive dental CAD, Exocad) based on digitized data and processed by computer assisted milling machine to specified dimension and then milled zirconia discs were sintered in sintering oven following the manufacturer's instructions as shown in figure 1. The six samples of monolithic and multilayer zirconia has been taken for sand blasting surface treatment. The samples were subjected to sandblasting with 50 μ m Al₂O₃ particles. This process was conducted at a steady distance of 10mm for 15 seconds at 0.2 MPa pressure. Following the procedure, the samples were thoroughly rinsed under water for 30 seconds and air dried to complete the preparation. For the Acid etching the six samples of both the groups has been taken. The samples underwent etching with 10% HF acid a duration of 60 seconds. After etching, they were rinsed with oil-free water and then air dried. A thin layer of silane coupling agent was applied to the samples and left undisturbed for 60 seconds.. This was followed by a gentle air blow for 5 seconds to finalize the process. The groups included are untreated monolithic zirconia, sandblasted monolithic zirconia, acid etch monolithic zirconia, untreated multilayered zirconia, acid etch multilayered zirconia and sandblasted multilayered zirconia. All the groups of the samples were subjected to surface roughness test. Surface roughness of the prepared samples was evaluated using a stylus profilometer SJ310 Mitutoyo with the diamond-tipped stylus as shown in figure 2. The profilometer is suitable and reliable instrument for evaluating surface roughness both qualitatively and quantitatively.

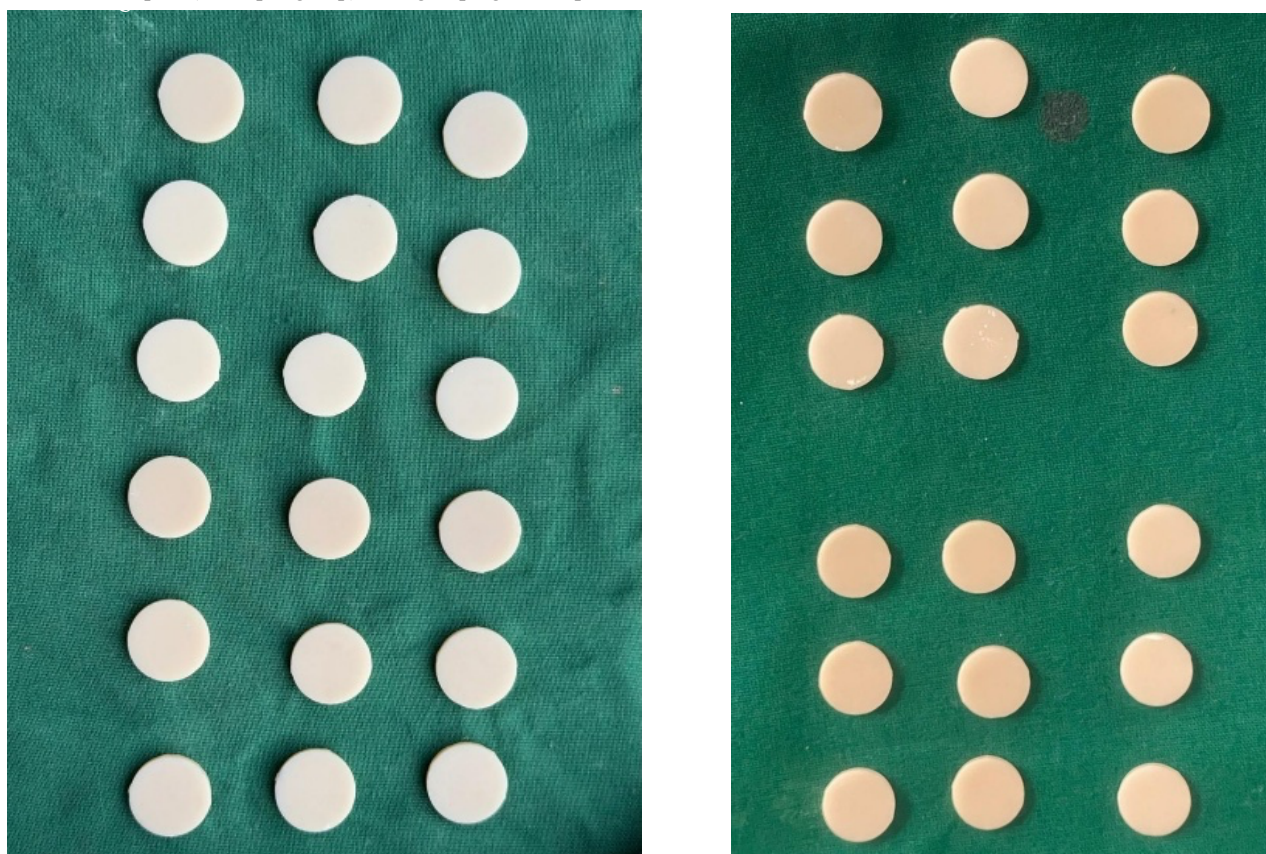


Figure 1: a. Monolithic Zirconia b. Multilayered Zirconia



Figure 2: Profilometer

RESULTS

The data obtained were analyzed using Statistical Package of Social science (SPSS; IBM Chicago Inc., USA). The significance level was set at $p \leq 0.05$ for the analysis. The statistical tests were selected based on the data type. All continuous data underwent the Kolmogorov Smirnov test for normality. The data was normally distributed ($p > 0.05$), thus allowing for use of

parametric tests of significance. Mean and standard deviations were calculated. The effect of various surface treatment methods on the surface roughness of monolithic and multilayered zirconia was compared by using a One -way analysis of variance. A post hoc tukey analysis was employed for multiple comparisons among the various methods.

Table 1: Surface roughness of monolithic and multilayered zirconia after different surface treatments.

Groups	Surface Roughness (μm)								
	N	Mean	Std. Deviation	Std. Error of Mean	Median	Minimum	Maximum	Range	
Monolithic zirconia	Untreated	6	0.436	0.29305	0.11964	0.4055	0.15	0.87	0.72
	Sandblasted	6	2.2858	0.61701	0.25189	2.3585	1.34	3.13	1.79
	Acid etch	6	0.4475	0.28581	0.11668	0.3995	0.18	0.8	0.62
Multilayered zirconia	Untreated	6	0.394	0.10696	0.04367	0.381	0.26	0.59	0.33
	Sandblasted	6	0.6178	0.18099	0.07389	0.6335	0.42	0.82	0.4
	Acid etch	6	0.3762	0.29795	0.12164	0.3555	0.03	0.86	0.83

Table 2: Intergroup comparison of monolithic zirconia with various surface zirconia specimens

	Mean Difference(I-J)	Std. Error	Sig.	95% Confidence Interval			
Dependent variable	(I) Group	(J) Group				Lower Bound	Upper Bound
Surface Roughness	Untreated monolithic zirconia	Sandblasted monolithic zirconia	-1.84983*	.19454	.001**	-2.4415	-1.2581
		Acidetch monolithic zirconia	-.01150	.19454	1.000	-.6032	.5802
		Untreated multilayered zirconia	.04200	.19454	1.000	-.5497	.6337
		Acidetch multilayered zirconia	-.18183	.19454	.934	-.7735	.4099
		Sandblasted multilayered zirconia.	.05983	.19454	1.000	-.5319	.6515

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	Mean Difference(I-J)	Std. Error	Sig.	95% Confidence Interval			
	Sandblasted monolithic zirconia	Untreated monolithic zirconia	1.84983*	.19454	.001**	1.2581	2.4415
		Acid etch monolithic zirconia	1.83833*	.19454	.001**	1.2466	2.4300
		Untreated multilayered zirconia	1.89183*	.19454	.001**	1.3001	2.4835
		Acidetch multilayered zirconia	1.66800*	.19454	.001**	1.0763	2.2597
		Sandblasted multilayered zirconia.	1.90967*	.19454	.001**	1.3180	2.5014
	Acid etch monolithic zirconia	Untreated monolithic zirconia	.01150	.19454	1.000	-.5802	.6032
		Sandblasted monolithic zirconia,	-1.83833*	.19454	.001**	-2.4300	-1.2466
		Untreated multilayered zirconia	.05350	.19454	1.000	-.5382	.6452
		Acid etch multilayered zirconia	-.17033	.19454	.949	-.7620	.4214
		Sandblasted multilayered zirconia.	.07133	.19454	.999	-.5204	.6630

*. The mean difference is significant at the 0.05 level.

Table 3: Intergroup comparison of multilayered zirconia with various surface zirconia specimens

	Mean Difference(I-J)	Std. Error	Sig.	95% Confidence Interval			
Dependent variable	(I) Group	(J) Group				Lower Bound	Upper Bound
Surface Roughness	Untreated multilayered zirconia	Untreated monolithic zirconia	-.04200	.19454	1.000	-.6337	.5497
		Sandblasted monolithic zirconia	-1.89183*	.19454	.001**	-2.4835	-1.3001
		Acidetch monolithic zirconia	-.05350	.19454	1.000	-.6452	.5382
		Acidetch multilayered zirconia	-.22383	.19454	.856	-.8155	.3679
		Sandblasted multilayered zirconia	.01783	.19454	1.000	-.5739	.6095
	Acid etch multilayered zirconia	Untreated monolithic zirconia	.18183	.19454	.934	-.4099	.7735
		Sandblasted monolithic zirconia	-1.66800*	.19454	.001**	-2.2597	-1.0763
		Acid etch monolithic zirconia	.17033	.19454	.949	-.4214	.7620
		Untreated multilayered zirconia	.22383	.19454	.856	-.3679	.8155
		Sandblasted multilayered zirconia	.24167	.19454	.813	-.3500	.8334
	Sandblasted multilayered zirconia.	Untreated monolithic zirconia	-.05983	.19454	1.000	-.6515	.5319
		Sandblasted monolithic zirconia	-1.90967*	.19454	.001**	-2.5014	-1.3180
		Acid etch monolithic zirconia	-.07133	.19454	.999	-.6630	.5204
		Untreated multilayered zirconia	-.01783	.19454	1.000	-.6095	.5739
		Acid etch multilayered zirconia	-.24167	.19454	.813	-.8334	.3500

*. The mean difference is significant at the 0.05 level.

The average values of surface roughness exhibited significant differences across the various surface treatment techniques and between groups of monolithic and multilayered zirconia.

Table 1 shows the comparison of the effect of various surface treatment methods on the surface roughness of monolithic and multilayered zirconia through One -way analysis of variance. The Surface Roughness was determined to be statistically significant, with p value 0.001 among different Groups. Table 2&3 shows

intergroup comparison of monolithic and multilayered zirconia with various surface zirconia specimens.

DISCUSSION

Surface roughness is the value of the texture on the surface of a material. It is evaluated by measuring the surface roughness (Ra) value[10]. Surface roughness (Ra) is a quantitative calculation of the relative roughness of a linear profile or area, was expressed as a single numeric parameter (Ra)[11]. Surface treatment of ceramics increases the surface area and creates microporosities available for the penetration of resin cement or veneering porcelain, enhancing the mechanical bonding. A strong resin bond relies on micromechanical interlocking and chemical bonding to the ceramic surface, which requires surface roughening and cleaning for adequate surface activation[12]. Surface roughness significantly affects the clinical performance of zirconia restorations, as it directly influences bonding efficacy, plaque accumulation, and long-term durability[13]. Surface treatment of zirconia can cause localized stress concentration and facilitate tetragonal-to-monoclinic phase transformation, which adversely affects the mechanical properties of zirconia, such as its flexural strength, hardness, and modulus of elasticity[14]. This research examined the effect of surface treatment methods on surface roughness of monolithic and multilayered zirconia. The null hypothesis was rejected, as a statistically significant difference in surface roughness was observed between monolithic and multilayered zirconia, particularly following sandblasting, indicating that surface treatment method influences the roughness characteristics of the two zirconia types differently. For monolithic zirconia, the sandblasted group exhibited the greatest surface roughness (mean = $2.29 \pm 0.62 \mu\text{m}$), indicating a marked increase in surface irregularities compared to other treatments. The untreated ($0.44 \pm 0.29 \mu\text{m}$) and acid-etched ($0.45 \pm 0.29 \mu\text{m}$) groups showed comparable and considerably lower roughness values, with minimal differences between them.

In the multilayered zirconia group, surface roughness values were generally lower than those observed in monolithic zirconia. The sandblasted specimens exhibited a moderate increase in roughness ($0.62 \pm 0.18 \mu\text{m}$) compared to the untreated ($0.39 \pm 0.11 \mu\text{m}$) and acid-etched ($0.38 \pm 0.30 \mu\text{m}$) groups, which showed similar mean values. Overall, sandblasting produced the greatest increase in surface roughness, particularly in monolithic zirconia. Acid etching showed no significant difference in surface roughness when compared with untreated samples for both zirconia types. Monolithic zirconia showed a more response to sandblasting than multilayered zirconia. Intergroup comparison revealed that the sandblasted monolithic zirconia group demonstrated significantly higher surface roughness than all other groups as shown in table 2. Monolithic zirconia has its homogeneous microstructure, appears to be more susceptible to surface alterations following airborne particle abrasion than multilayered zirconia, which consists of layers with varying translucency and composition. Khaled Q in the year 2018 concluded that Polishing resulted in a significant improvement in the surface roughness of both monolithic and layered zirconia restorations[15]. Kern and Wegner and Kosmac et al in the year 1999 reported that sandblasting produces pronounced surface irregularities by creating micro-retentive features, which explains the significantly higher roughness observed in the sandblasted groups of the present study, particularly in monolithic zirconia[16,17]. Similar observations were made by Hallmann et al in the year 2010 showed that sandblasting significantly alters zirconia surface topography, while acid etching showed minimal surface changes[18]. Kim et al and Akin et al have shown that untreated zirconia surfaces have poor bonding strength due to their smoothness and inert surface characteristics[19]. The significantly higher surface roughness observed in the sandblasted zirconia groups can be attributed to the mechanical action of airborne-particle abrasion. During sandblasting, high-velocity alumina particles impact the zirconia surface, causing micro-cutting, plastic deformation, grain pull-out, and the formation of irregular pits and grooves. These alterations increase

the surface area and create micro-retentive features, resulting in higher Ra values. In contrast, untreated zirconia surfaces remain relatively smooth because of the dense polycrystalline structure and absence of surface modification. Acid etching demonstrated minimal effect on surface roughness because zirconia lacks a glassy silica phase that is susceptible to acid dissolution. Acid-etched zirconia exhibited roughness values comparable to those of untreated specimens. Previous studies have suggested that a surface roughness (Ra) value ranging from 0.2 to 0.5 μm is generally considered clinically acceptable for zirconia restorations, as it minimizes plaque accumulation and reduces wear of the opposing dentition while maintaining favorable surface characteristics[20]. While increased surface roughness is desirable for micromechanical retention and resin cement bond strength, a very high surface roughness may lead to adverse clinical effects such as increased wear of opposing dentition and potential compromise of the integrity of zirconia structure [21]. This study had limitations as only two surface treatment methods were evaluated with untreated controls, while other methods such as laser treatment, selective infiltration etching, and polishing were not included. Additionally, only surface roughness (Ra) was measured, and other important properties like bond strength, phase transformation, flexural strength, wear, and aging resistance were not assessed. The study also used zirconia from a single manufacturer, and differences in composition, processing in other brands may affect the outcomes. Future research should include thermocycling, mechanical loading, and aging to better simulate oral conditions, and use advanced techniques such as SEM, AFM, and XRD to more thoroughly analyse surface changes of zirconia.

CONCLUSION

Based on the limitations of this in vitro study, it can be concluded that surface treatment methods significantly influence the surface roughness of zirconia restorations. Sandblasting significantly increased surface roughness, with monolithic zirconia showing greater changes than multilayered zirconia, while acid etching had minimal effect. Surface treatment plays a crucial role in improving the bonding performance of zirconia restorations. These findings suggest that surface treatment protocols should be chosen according to the type of zirconia to improve bonding, durability, and clinical performance

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CONFLICT OF INTEREST

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