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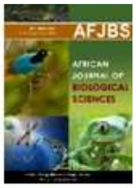

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Nanostructured Surface Modification of Zirconia Using TiO_2 Nanotubes: An Evaluation of Surface Roughness

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Abstract

Objectives: To evaluate the effect of various surface pretreatment protocols on surface roughness characteristics of zirconia ceramics.

Methods: TiO_2 nanotubes were synthesized by alkaline hydrothermal method. Twenty zirconia disc specimens (5 mm in diameter × 3 mm thick) were prepared and randomly divided into two groups (n=10): sandblasting with Al_2O_3 (Group I); TiO_2 nanotubes deposition (Group II). Surface roughness (Ra, µm) was measured using a contact profilometer under standardized conditions. Data were statistically analyzed using independent-samples t-test.

Results: Statistical analysis revealed that the mean surface roughness of Group II (0.97) was higher than that of Group I (0.68), with a statistically significant difference between the groups ($p < 0.001$).**Conclusions:** Within the limitations of the present study, TiO_2 nanotubes surface modification significantly enhanced zirconia surface roughness.

Keywords: Zirconia, TiO_2, Surface roughness, Shear bond strength.

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Introduction

Zirconia ceramics (zirconium dioxide, ZrO_2) have become a widely adopted material in modern dentistry owing to their desirable esthetic characteristics, high fracture resistance, and remarkable mechanical stability.***(Denry and Kelly, 2008; Zhang and Lawn, 2018)*** These favorable characteristics make zirconia an appropriate material for indirect restorations, including crowns, fixed dental prostheses, and implant-supported prosthetic reconstructions. However, reliable and durable bonding to zirconia is still considered a major challenge.***(Siddiqi et al., 2017)***

Zirconia differs from silica-based ceramics in that it is a polycrystalline material lacking a glassy matrix, which limits the effectiveness of conventional acid etching and silane-based bonding techniques. Consequentely, effective adhesion primarily relies on surface modification strategies that enhance surface roughness and surface energy, thereby improving micromechanical retention and facilitating chemical interaction with resin-based materials.***(Komine et al., 2010; Al-Amari et al., 2024)***

Numerous surface treatment strategies have been proposed, including airborne-particle abrasion, tribochemical silica coating, selective infiltration etching, laser irradiation, plasma treatment, and the use of functional primers.***(Mohamed et al., 2025)*** Among these techniques, airborne-particle abrasion with aluminum oxide particles remains the most frequently used in clinical practice due to its simplicity and its ability to produce immediate surface roughening. However, concerns have been raised regarding its potential to introduce surface defects, induce phase transformation, and compromise the long-term structural integrity of zirconia restorations.***(Kern and Wegner, 1998; Senyilmaz et al., 2007; Papia et al., 2014)***

In addition to mechanical surface modification, chemical strategies have been developed to improve adhesion between zirconia and resin materials. Combining mechanical pretreatments with primers or resin cements containing functional phosphate monomers, most notably 10-methacryloyloxydecyl dihydrogen phosphate (10- MDP), has been shown to enhance bonding effectiveness by integrating micromechanical interlocking with chemical interaction.***(Aboushelib et al., 2008; Inokoshi et al., 2014; Yang et al., 2018; Alammar et al., 2022)*** The phosphate group of 10-MDP able to form stable ionic hydrogen bonds with zirconium oxide. At the same time surface roughening increases both surface area and surface energy, which together contribute to improved bond durability. However, the effectiveness of these chemo-mechanical approaches has been reported to vary, and degradation of the bonded interface after aging remains a concern.***(Lehmann and Kern, 2009; Kern, 2015)***

More recently, nanostructured surface modification has been proposed to optimize zirconia surface characteristics. Titanium dioxide (TiO_2) nanotubes have gained interest due to their high surface area, chemical stability, and compatibility with both ceramic substrates and resin materials.***(Arun et al., 2023; Padmavathy et al., 2024)*** Therefore, this study aimed to evaluate the influence of different surface pretreatments on zirconia surface roughness. The null hypothesis was that surface treatment would not significantly affect zirconia surface roughness.

Materials and Methods

The materials used in this study are listed in **Table 1.**

Table 1. Materials used in the present study.

Material	Composition	Manufacturer	Lot. No
TiO_2 Anatase Powder	Titanium dioxide (anatase phase)	Sigma-Aldrich, USA	MKBP2332V
Isopropyl Alcohol	2-Propanol (C_3H_8O, ≥99.5%, analytical grade, solvent)	Sigma-Aldrich, USA	SHBG4888V
Monolithic Dental Zirconia Ceramic Blocks	High translucent zirconia (Y-TZP, Yttria-stabilized tetragonal zirconia polycrystal)	KINGCH, China	24013103229812
Al_2O_3 Powder	50 µm Al_2O_3 airborne particles	Korox®, BEGO, Germany	15712950314

Synthesis of TiO_2 nanotubes

The TiO_2 nanotubes were synthesized using hydrothermal method as described by Arruda et al.***(Arruda et al., 2015)***

Sample size calculation

Based on prior surface roughness data (Abu-Eittah, 2012)***(Abu-Eittah, 2012)***, using G*Power software (version 3.1.9.7) and assuming an effect size of 0.98 with α = 0.05, 10 specimens per group provided sufficient statistical power 95% to detect significant differences among two groups.

Specimen Preparation and Grouping

Monolithic zirconia blocks were sectioned into disc-shaped specimens (5 mm diameter × 3 mm thickness) using a CAD/CAM milling machine (Imes

Icore 150i Pro, Germany). Twenty specimens were randomly allocated into two experimental groups (n = 10) based on surface treatment:

- **Group I:** Al_2O_3 sandblasting
- **Group II:** TiO_2 nanotubes deposition

Surface Treatment Procedures

Group I: Air abrasion with 50-µm Al_2O_3 particles from 10 mm distance at 45° angle, 0.2 bar for 10 s.***(Kim and Ahn, 2021)***

Group II: TiO_2 nanotubes dispersed in isopropyl alcohol were applied to presintered zirconia under high-pressure vacuum for uniform deposition.***(Mezarina-Kanashiro et al., 2022)***

Drying and Sintering of Zirconia Specimens

All specimens were dried at 37 °C for 24 h and sintered in a high-temperature furnace (TABEO-1/M/ZIRKON-100, Nabertherm, Germany) following the manufacturer's fast-sintering protocol.***(Mezarina-Kanashiro et al., 2022)***

Surface Roughness Analysis using Profilometer Analysis (Ra)

Surface roughness was measured using a contact profilometer (Mitutoyo SJ-210, Kawasaki, Japan). A conical diamond stylus traced the surface to generate a 2D profile, following ISO 21920-2, ISO 21920-3, and ISO 3274 standards.***(Buchenau et al., 2023; Sushil et al., 2025)*** Measurements were taken at a cut-off of 0.25 mm and speed of 0.5 mm/s, calibrated with a reference block (Ra = 2.792 µm). Five measurements were averaged for each specimen.***(Rosentritt et al., 2024)***

Statistical Analysis:

Statistical analysis was performed using an independent-samples t-test to compare the mean surface roughness values between the two groups.

Results

Means, standard deviations and Independent Samples t-test results of surface roughness for different groups are shown in (Table 1) and presented graphically in (Figure 1). The mean value of Group II (TiO_2 Nanotubes)(0.97) was higher than the mean value of Group I (Sandblasting) (0.68). There was a significant difference in surface roughness between different study groups ($p < 0.001$).

Table 1: Means, standard deviations and Independent Samples t-test of surface roughness of study groups.

Study Groups	Group I (Sandblasting)	Group II (TiO_2 Nanotubes)	p-value
Mean ± SD	0.68 ± 0.11	0.97 ± 0.15	< 0.001*

*Statistically significant difference at $P < 0.05$

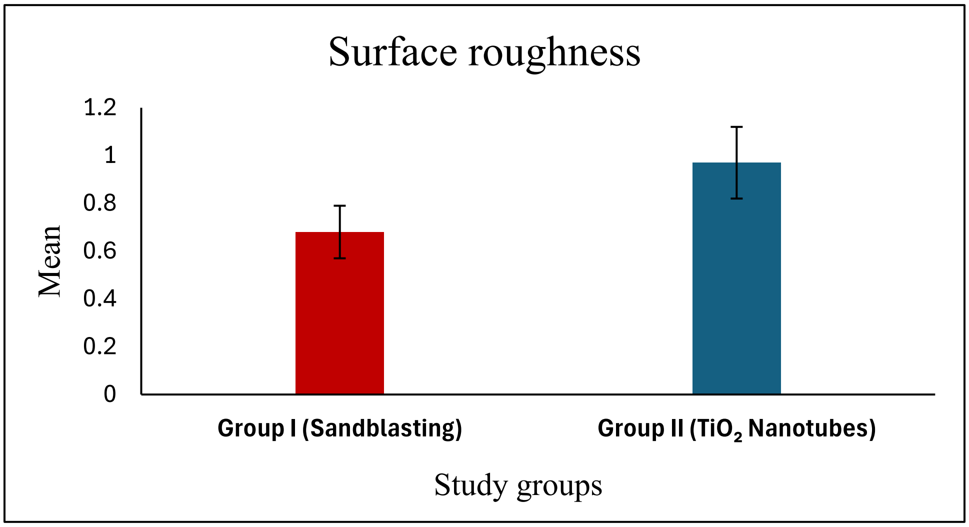

Figure 1. Bar chart representing the mean values of surface roughness of study groups.

Discussion

Zirconia is widely employed as a core material for metal-free indirect restorations due to its high strength, fracture resistance, and compatibility with CAD/CAM fabrication.***(Zhang and Li, 2024; Gheorghe and Adela, 2025; Melnyk et al., 2025)*** However, durable bonding to zirconia remains more technique-sensitive than bonding to silica-based ceramics, largely due to its polycrystalline, non-etchable structure and limited surface reactivity.***(Reddy et al., 2014; Dos Santos et al., 2018; Quigley et al., 2021)***

Surface roughness is the key factor in zirconia bonding because it directly influences surface area, wettability, and the ability to achieve micromechanical interlocking.***(Wongsue et al., 2023)*** In the present study, surface roughness was measured using a contact stylus profilometry, which provides standardized and reproducible two-dimensional measurements in accordance with ISO specifications.***(Standardization, 2021; Rosentritt et al., 2024)*** Although this method cannot identify undercut or re-entrant features, it is still considered a clinically relevant technique for comparing surface characteristics.***(Haitjema and Leach, 2018; Buchenau et al., 2023)***

The results showed that the applied surface treatment had a significant effect on surface roughness. The higher Ra values were recorded in Group II (TiO_2 nanotubes), whereas sandblasting alone (Group I) resulted in lower surface roughness. These findings suggests that nanostructured surface treatment modification is more effective than the conventional mechanical roughening in changing the surface topography of zirconia.***(Öztemel et al., 2025)***

The increased roughness observed in the TiO_2 groups may be related to their nanotubular structure, which creates nanoscale surface irregularities and increase the available surface area.***(Patel et al., 2017)*** Accordingly, Group II (TiO_2 nanotubes) exhibited higher roughness values, indicating that nanotube deposition can modify the zirconia surface.

Overall, these findings confirm that nanostructured TiO_2 surface modification, results in higher increase in zirconia surface roughness, which could markedly impact subsequent bonding performance.

Conclusion

Within the limitations of this study, zirconia surface roughness was significantly affected by the type of surface treatment applied. The TiO_2 nanotubes yielded higher roughness, whereas sandblasting alone produced lower values. These findings suggest that nanostructured surface modification may serve as an effective strategy for improving zirconia surface properties prior to adhesive procedures.

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