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Evaluation the effect of glazing and the printing orientations on the surface roughness and the microhardness of DLP dental zirconia: an in vitro study

Avaliação do efeito da aplicação do glaze e das orientações de impressão na rugosidade de superfície e na microdureza de zircônia odontológica DLP: um estudo in vitro

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ABSTRACT

Objective: To evaluate the effect of glazing and different printing angulations on the surface roughness and hardness of 3D-printed zirconia specimens. **Material and Methods:** Forty-two cuboid-shaped specimens (10mm length, 10mm width, 3mm thickness were constructed by ZIPRO printer (AON, South Korea) from a zirconia slurry. Three distinct groups were established for the specimens based on the printing orientation angle (n = 14), Vertical (0°), Horizontal (90°), and Diagonal (45°) orientations to the building direction. The diamond-impregnated system was used to polish all specimens as the manufacturer's recommendations. Two groupings were subsequently formed from the specimens (n =7): polished groups (VP, HP, and DP), polished and then glazed with the diamond paste groups (VG, HG, and DG). The surface roughness of each specimen was measured using a profilometer, and the microhardness was determined using a Vicker microhardness tester. For assessing the specimens' surface quality, the scanning electron microscopy apparatus was employed. One-way ANOVA and Tukey's HSD tests were used in analyzing the study data, which had a significant P-value ($p < 0.05$). **Results:** A significant difference was observed between the polishing and glazing groups for surface roughness and microhardness; however, no significant differences were identified in surface roughness between the polished and glazed groups in horizontal and diagonal orientations. **Conclusion:** Glazing with diamond paste improved the zirconia surface qualitatively and quantitatively. Hardness values were increased in the glazed groups compared to the polished groups in all three building orientations. The optimal building angulation was the vertical orientation.

KEYWORDS

Additive manufacturing; Digital light processing zirconia; Glazed 3D-printed zirconia; Microhardness; Surface roughness.

RESUMO

Objetivo: Avaliar o efeito da aplicação do glaze e de diferentes angulações de impressão na rugosidade de superfície e na dureza de espécimes de zircônia impressos em 3D. **Material e Métodos:** Quarenta e dois espécimes em formato cuboide (10 mm de comprimento, 10 mm de largura, 3 mm de espessura foram coneccionados pela impressora ZIPRO (AON, Coreia do Sul) a partir de uma pasta de zircônia. Três grupos distintos foram estabelecidos para os espécimes com base no ângulo de orientação de impressão (n = 14), orientações Vertical (0°), Horizontal (90°) e Diagonal (45°) para a direção da impressão. O sistema impregnado com diamante foi usado para polir todos os espécimes conforme as recomendações do fabricante. Dois grupos foram posteriormente formados a partir dos espécimes (n = 7): grupos polidos (VP, HP e DP), grupos polidos e glazeados com pasta de diamante (VG, HG e DG). A rugosidade da superfície de cada espécime foi medida usando um perfilômetro, e a microdureza foi determinada usando um aparelho de microdureza Vicker. Para avaliar a qualidade da superfície dos espécimes, o equipamento de microscopia eletrônica de varredura foi utilizado. Testes ANOVA unidirecional e teste Tukey HSD foram utilizados na análise dos dados do estudo, que tiveram um valor P significativo ($p < 0,05$). **Resultados**: Foi observada uma diferença significativa entre os grupos de polimento e glaze na rugosidade da superfície e microdureza; no entanto, nenhuma diferença significativa foi identificada na rugosidade da superfície entre os grupos polido e glazeados nas orientações horizontal e diagonal. **Conclusão**: A aplicação do glaze com pasta de diamante melhorou a superfície da zircônia qualitativa e quantitativamente. Os valores de dureza foram aumentados nos grupos glazeados em comparação aos grupos polidos em todas as três orientações. A angulação ideal da impressão foi a orientação vertical.

PALAVRAS-CHAVE

Manufatura aditiva; Zircônia de processamento por luz digital; Zircônia glazeada impressa em 3D; Microdureza; Rugosidade de superfície.

INTRODUCTION

Additive manufacturing (AM) has surpassed traditional methods in manufacturing for its fundamental shape-creation capabilities, design flexibility, waste minimization, and capacity to be customized. This technique employs a printing process that builds upon itself, resulting in products with long-lasting mechanical properties [1]. The Organization for Economic Cooperation and Development (OECD) predicts that AM will ultimately supersede Subtractive Manufacturing (SM) shortly [2]. Zirconia restorations are predominantly produced through milling utilizing a CAD-CAM process. The introduction of lithography-based ceramic manufacturing (LCM) occurred a few years ago [3]. Zirconia ceramics provide excellent mechanical properties, including strength, hardness, fracture toughness, wear resistance, corrosion resistance, and biocompatibility, while facilitating machining by SM technology in the pre-sintering phase [4]. Vat photopolymerization, material jetting, sheet lamination, powder bed fusion (PBF), material extrusion or fused deposition modeling, binder jetting, and direct energy deposition are the seven AM approaches categorized by the American Society of Testing and Materials [5,6]. Promising outcomes for generating zirconia-based dental prostheses have been established by stereolithographic techniques, like Stereolithography (SLA) and Digital Light Processing (DLP), thanks to the advent of AM technology [7,8]. Recent investigations indicate that vat photopolymerization when accompanied by appropriate heat treatment techniques, can attain a density and microstructure comparable to those of conventional manufacture [9]. The mechanical properties of the AM zirconia are close to those of SM blocks, including high strength, hardness, and fracture toughness, all of which are influenced by the powder combination [8,10]. One of the factors that, when adjusted properly, decreases printing duration and manufacturing cost while increasing the dimensional precision of the final product is the printing orientation [11]. Yet, there are still problems with 3D-printed ceramics' performance, such as the fact that different printing layer orientations produce materials with varying characteristics. According to previous investigations, zirconia specimens printed with layers orientated vertically to the tensile surface exhibited reduced flexural strength and elastic modulus in bending tests compared to specimens printed with layers aligned to the tensile surface [12,13]. So, to find out what printed materials are suitable for use and to design the orientation of printed restorations for buildings, it's crucial to know how different printing layer directions affect their mechanical characteristics and longevity under fatigue. Surface quality is an additional obstacle for 3D-printed dental ceramics along with mechanical behavior as a result of the step effect brought about by building in layers [14]. In its as-sintered state, 3D-printed zirconia displays significant surface roughness, especially on printing-characterized surfaces [15]. Despite using the same polishing procedures, mechanical properties showed that surface roughness was the sole distinguishing feature between the vertical and horizontal specimens [16]. Prior research indicated that construction direction can affect the surface roughness of printed zirconia because of the creation of tiny grooves at layer borders during 3D printing [17]. The profiles produced at low print orientation angles are regular, with the peak amplitude directly proportional to the height of the layers. The width of the peaks widens with steep print orientation angles, while the flat area or gap between successive peaks remains the same. The experimental data demonstrate a more progressive decline starting at approximately 85 degrees, whereas the simulated roughness would be zero at 90 degrees because the direction of measurement coincides with the direction of the printed layers [18]. Increased surface roughness may contribute to more bacterial adhesion and worse tooth/restoration deterioration, each of those being undesirable for dental restorations [19]. Crack propagation under cyclic loading can be initiated by a surface defect, which in turn affects the fatigue life and material strength [20]. Zirconia restorations produced with AM technology are still in their early stages of marketing, thus products have yet to be widely used in the clinic [21]. Research has shown that polishing ground zirconia can reduce surface roughness just as well as glazing [22,23]. Meanwhile, some research has shown that glazing is the most effective method for getting a smooth surface [24,25]. To what extent would printing layer orientation and glazing affect the topography, surface roughness, and microhardness of 3D-printed zirconia produced by a vat photopolymerization DLP printer? That is what the study was primarily meant to achieve. The study hypotheses were: (1) the different printing orientations will not significantly impact the surface roughness and microhardness, and (2) glazing will significantly impact the surface roughness and hardness of the DLP zirconia specimens.

MATERIAL AND METHODS

Specimen grouping

A cuboid-shaped specimen with 10 mm length,10 mm width, and 3 mm [26] thickness was designed by using an open-source specialized 3D modeling program (Exocad, Dental DB, 3.0 Galway, Darmstadt, Germany), and an STL file was composed. Under the building angulation, the specimens were categorized into three groups, with fourteen specimens in each one:

- V Group: The first group consisted of 14 Vertical orientation specimens fabricated at 0° angulation parallel to the building direction.
- H Group: The second group consisted of 14 Horizontal orientation specimens fabricated at 90° angulation perpendicular to the building direction.
- D Group: The third group consisted of 14 Diagonal orientation specimens fabricated at 45° angulation to the building direction as shown in Figure 1.

Based on the surface treatment, each was split into two categories, with seven specimens in each:

- (VP, HP, and DP) polished by a diamond polishing system.
- (VG, HG, and DG) polished by a diamond polishing system and then glazed with a diamond paste.

Specimens' fabrication

The STL file needs to be sliced by the dental software program (ZIPROS slicing software AON Co., Ltd), and support structures were added. Forty-two specimens were printed in three building directions (V, H, and D) by a DLP printer (ZIPRO, AON Inc., Seoul, South Korea) from the zirconia slurry (INNI-CERA BCM-W500/1000, ZIPRO Dental, AON Inc., South Koria), with 50 μm layer thickness, and 10000 μw /cm^2 light intensity as the recommendations of the manufacturers. Table I shows the chemical composition of the zirconia slurry used according to the manufacturer. After

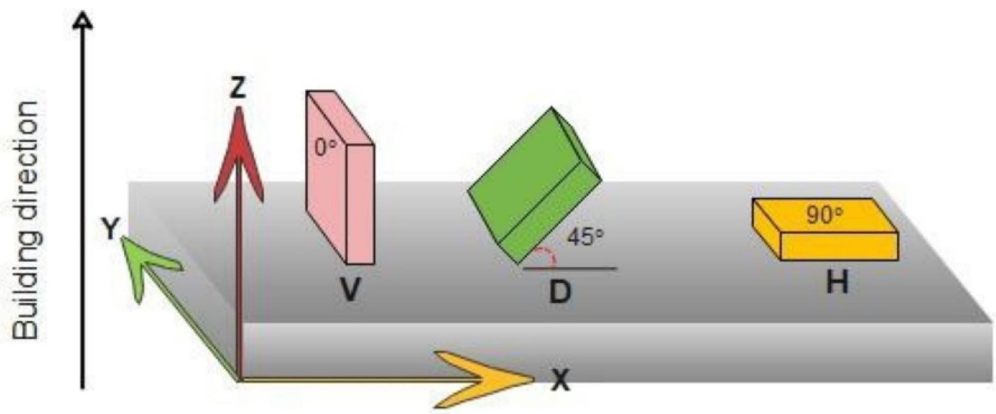

Figure 1 - Specimens' angulations

Table I - Chemical composition of 3D printer zirconia specimens used in this study

	Material	Manufacturer	Composition	Lot. No.
Additive Manufacturing (AM) DLP printer zirconia	Dental Zirconia Material, INNI-CERA BCM-W1000 slurry	ZipPro Dental AON Co., Ltd. South Koria	The photoinitiator, Monomer, Oligomer, additives, and 3mol% yttria-partially stabilized tetragonal zirconia polycrystals (3Y-TZP).	A1231013-003

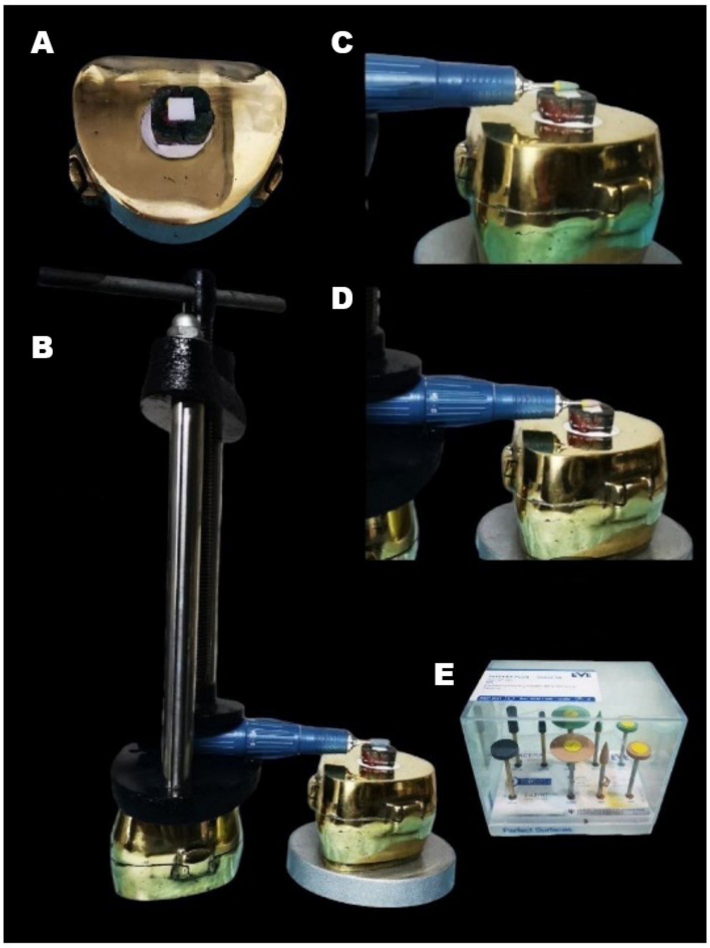

Figure 2 - A: the custom-made holder contained the specimens mounted in a dental flask, B: the pre-polishing process by DYP-13 g bur, C and D: the polishing process by H2DCmf bur, H2DC bur respectively, E: the diamond impregnated system.

the printing procedure was complete, the printed specimens' support structures were carefully removed during the green stage using a sharp blade [27], and isopropanol (purity ≥ 99.5%) was used for cleaning the specimens according to the manufacturer's instructions. Specimens were then de-binded and sintered in a 3D-printed zirconia sintering furnace (ZIRFUR, AON, South Koria) at 1500 Cº for 21h and 45 min with a heating rate of 0.2-10 Cº/h according to the manufacturing instructions [3]. All specimens were polished to a mirror shine to ensure the surfaces were uniformly flat. A cubic custom-made self-cure acrylic holder (2 cm × 2 cm × 3 cm) with a middle hole used for placing the specimen and having two cross-shaped groves for easy removal after completion. This holder was made to facilitate the handling of the specimens during treatment procedures. The holder contains the specimen mounted in a dental flask as shown in Figure 2A. The pre-polishing process was performed using a diamond-impregnated system DIACERA Figure 2E in one direction for 60 s to the entire surfaces as in Figure 2B. The polishing process was performed using DIASYNT in two stages, firstly, by using green, medium grit at a speed of 7000 to 12000 rpm in one direction for 60 seconds to the entire surface. Secondly, high-gloss polishing was carried out using pink, fine grit at 4000 to 8000 rpm in one direction for 60 s to the whole surface as in Figure 2C and D. So, the high polish keeps the antagonist from getting scratched up. A single bur was used to polish every seven specimens. After polishing, each specimen was measured using an electronic digital caliper (INSIZE®, China) to guarantee precision. The process is carried out following the manufacturer's requirements. Seven specimens of each subgroup were glazed by applying a uniform thickness of glazing diamond paste (HeraCeram glaze universal 20g from KULZER, Hanau, Germany). The glazing paste was thoroughly mixed, and each specimen was positioned on the holder and uniformly coated with a fine zirconia brush (MPF BRUSH Co., Cyprus). The specimens were then sintered at (850 C°) in the ceramic sintering furnace (VITA VACUMAT® 6000 M, Zahnfabrik, Germany) for 10 min according to the recommended firing program.

Surface roughness (Ra) test

A portable roughness tester TR220 was employed to quantitatively measure the surface roughness of all specimens in micrometers (μm). Each specimen had its mean roughness profile (Ra) determined; so, the total surface roughness could be described. The average values of each specimen were determined by taking three readings at different locations [28].

Vicker microhardness measurements

All specimens were measured using a Vickers microhardness tester machine. The specimens are placed on the horizontal stage of the tester and subjected to a force of 9.8 N (1k) for 15 seconds using a diamond Vickers indenter. The optimal Vickers indenter is a perfectly polished, pointed, square-based pyramidal diamond with face angles of 136°. The specimen size is often a minimum of 0.50 mm and exceeds 10 times the indentation depth (ASTM C1327-2015) [29]. The subsequent expression computes the Vickers hardness:

$$HV = \alpha \, P/d^2 \quad (1)$$

Where HV represents the Vickers hardness value in kgf/mm^2, P denotes the applied force in kg, d represents the mean value of the indentation diagonals in mm, and α is the indenter's geometric constant, equivalent to 1.8544 according to (ASTM E384-22) [30]. The hardness value of every sample can be automatically determined through the tester devices.

Scanning Electron Microscopy (SEM) test

The SEM was utilized to assess the surface topography, one specimen was randomly selected from each group. After cleaning the specimens, gold nanoparticles (24 carats) were applied to their surface by using a Plasma Scattering Coater Device. Then, they were clamped onto the SEM specimen holder and placed in the scanning electron microscope's chamber. The SEM photomicrographs were captured at 250 X magnification, having a working distance of 400 μm, and an acceleration voltage of 30.00 Kev [31].

Statistical analysis

The social science statistics package SPSS version twenty-four was used to examine the research data. The Shapiro-Wilk test was conducted to assess normality. Levene's test was employed to evaluate the homogeneity of variances. There was no violation of the homogeneity criteria, and the data followed a normal distribution. A one-way analysis of variance ANOVA with Tukey's HSD test was used for the statistical analysis. The recorded surface roughness and microhardness were averaged, and the standard deviations (SD) and the standard error were computed for each grouping. Considering statistical significance, P values less than 0.05 were considered.

RESULTS

Surface roughness test

Table II presents the descriptive statistics of the surface roughness values for polished and glazed groups in three building orientations of the specimens, including means, standard deviation, standard error, and confidence intervals for the mean, minimum, and maximum values.

As in Table II, the (Ra) value of the polished specimens was highest in the vertical orientation group and lowest in the horizontal orientation group. For the glazed groups, the (Ra) value was the highest in the diagonal orientation group and the lowest in the horizontal orientation group. F-test by using one-way ANOVA was done to identify if there had been a statistically significant variation in the mean values when comparing groups as in Table III. Tukey's HSD test was performed to determine the honest significant differences between groups as shown in Table IV.

Table II - Descriptive statistics of surface roughness (Ra) of polished and glazed zirconia specimens

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
RA VP	7	3.4040	.35586	.13450	3.0749	3.7331	2.90	3.86
RA HP	7	1.5807	.05989	.02264	1.5253	1.6361	1.50	1.68
RA DP	7	3.0153	.15347	.05801	2.8734	3.1572	2.83	3.28
RA VG	7	1.3909	.02509	.00948	1.3677	1.4141	1.34	1.42
RA HG	7	1.5869	.05509	.02082	1.5359	1.6378	1.52	1.64
RA DG	7	3.0650	.31984	.12089	2.7692	3.3608	2.72	3.55

Table III - One-way ANOVA between all groups (surface roughness test).

	Mean Square	F	P-value	Sig
Between Groups	5.822	134.484	0.000	*HS

***P < 0.001 High significant

Table IV – Tukey's HSD test for surface roughness of all groups

(I) Groups	(J) Groups	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
RA VP	RA HP	1.82329	.11577	.000	1.5039	2.1426
	RA DP	.38871	.11577	.013	.0694	.7081
RA HP	RA DP	-1.43457	.11577	.000	-1.7539	-1.1152
RA VG	RA HG	-.19600	.08720	.139	-.4366	.0446
	RA DG	-1.67414	.08720	.000	-1.9147	-1.4336
RA HG	RA DG	-1.47814	.08720	.000	-1.7187	-1.2376

The Vickers microhardness test

The descriptive statistics of the Vickers microhardness measurement for polished and glazed specimens in three building orientations including means, standard deviation, standard error, and confidence interval for the mean, minimum, and maximum values were presented in Table V. As in Table V, the microhardness value of the polished specimens was the highest in the vertical orientation group and the lowest in the horizontal orientation. For the glazed group, the microhardness value was the highest in the vertical orientation group and the lowest in the horizontal orientation group. Table VI presents the results of the ANOVA test employed to ascertain if the means of the groups exhibited significant differences. As demonstrated in Table VII, Tukey's HSD test was conducted to identify the significant differences among the groups.

SEM analysis

The representative images obtained from SEM in Figure 3a showed that the vertical orientation polished specimen had a layered strand pattern with a flat indentation across them, in a range of 178-191 nm. After glazing, the SEM image shows a good glazing process occurred. However, dark spots could result from gases escaping from the micro gaps between the layers as in Figure 3d.

The SEM measurement of the horizontal orientation specimens before glazing showed that the surface was a single plate with a surface containing depressions and elevations with a diameter of 1.3-2.4 μm, all of which were on one side as in Figure 3b. After glazing it was shown that a good glazing occurred with very few particles on the surface that did not exceed 2 μm in diameter as shown in Figure 3e.

Table V - Descriptive statistics of microhardness (VHN) zirconia polished and glazed groups in kgf/mm^2

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
VHN VP	7	1.7114	.00660	.00250	1.7053	1.7175	1.70	1.72
VHN HP	7	1.4619	.00558	.00211	1.4567	1.4670	1.46	1.47
VHN DP	7	1.6054	.01652	.00624	1.5901	1.6207	1.58	1.62
VHN VG	7	2.5911	.06473	.02447	2.5313	2.6510	2.49	2.69
VHN HG	7	1.9990	.04930	.01863	1.9534	2.0446	1.95	2.10
VHN DG	7	2.0647	.12755	.04821	1.9468	2.1827	1.90	2.20

Table VI - One-way ANOVA test between all groups (microhardness test)

	Mean Square	F	P-value	Sig
Between Groups	1.160	299.572	0.000	*HS

*P < 0.001 High significant

Table VII - Tukey's HSD test for microhardness of all groups

(I) Groups	(J) Groups	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
VHN VP	VHN HP	.24957	.00510	.000	.2355	.2636
	VHN DP	.10600	.00510	.000	.0919	.1201
VHN HP	VHN DP	-.14357	.00510	.000	-.1576	-.1295
VHN VG	VHN HG	.59214	.04442	.000	.4696	.7147
VHN DG	.52643	.04442	.000	.4039	.6490	
VHN HG	VHN DG	-.06571	.04442	.465	-.1882	.0568

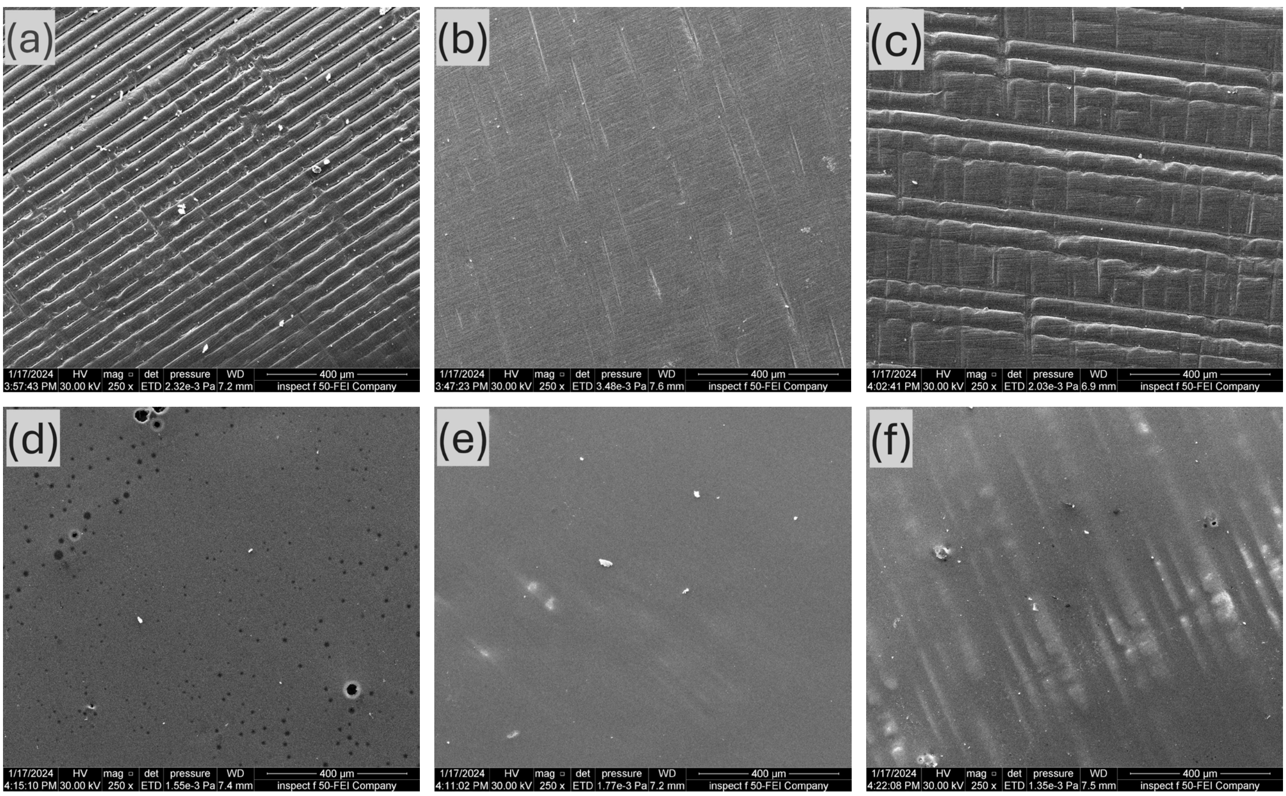

Figure 3 - SEM of 3D-printed zirconia showing (a) vertically polished, (b) horizontally polished, (c) diagonally polished, (d) vertically glazed, (e) horizontally glazed, (f) diagonal glazed specimens.

The SEM analysis of the diagonal orientation revealed the existence of plate-like structures in three different dimensions as in Figure 3c. After the glazing process, Figure 3f shows that a regular glazing process occurred with some light areas appearing on the surface which are attributed to the surface structure containing several plates with different heights.

DISCUSSION

This study examined the surface roughness and the microhardness of 3D-printed zirconia processed by a DLP printer in three building orientations during polishing and after the glazing process. The results of the recent study suggested the complete rejection of the first hypothesis because of highly significant differences in surface roughness and microhardness values between the different orientations of the AM zirconia specimens. The second hypothesis was partially rejected because the horizontal and diagonal orientations were not significantly affected by the glazing.

The accepted surface roughness for the dental restoration threshold is 0.2 μm, so an unpolished surface with a value higher than this may increase the propensity for bacterial adhesion and oral biofilm development [17]. The appropriate surface polishing protocols were performed in this study to improve the surface quality [17]. Mirror polishing using appropriate equipment and supplies that contain fine diamond particles is currently the suggested approach for zirconia surface finishing [32].

When comparing P specimens, the recent study revealed that the highest surface roughness value was in group RA VP, and the lowest was in group RA HP. These findings are in agreement with the study of Abualsaud et al. [33], they demonstrated that, in contrast to horizontal specimens, those with a vertical or diagonal orientation had a higher surface roughness due to differences in the orientation of roughness measurement concerning layer direction and the existence of steps between successive layers. These results are in harmony with the study of Schiltz et al. [16], they attributed the increase in surface roughness of the vertical orientation group to the surface topography of these two orientations (the vertical and the horizontal).

Also, the results of this study concurred with the study of Xing et al. [34], they studied the effect of printing orientations of 3D-printed zirconia on the horizontal and the vertical surfaces of the same specimen in different printing angulations, they found that the surface roughness value of the horizontal surface was lower than that of the vertical surface. When zirconia restorations are glazed, they create a uniform surface that is smooth and uniform in shape, protecting the antagonist tooth enamel and reducing the accumulation of plaque on the restoration [32].

The recent study results demonstrated a notable reduction in surface roughness of the horizontal orientation group that occurred after glazing. The highest surface roughness value was in the RA DG group, and the lowest was in the RA VG. Table III showed significant differences between the vertical orientation specimen P and PG groups ($P < 0.001$). Whereas, other orientation groups did not show any significant differences between the P and PG specimens. The SEM analysis images validated these findings on the surface roughness (Figure 3 d, e, f). This improvement in surface topography after applying the diamond paste is consistent with Branco et al. [35] they demonstrate that the glaze covering makes the surface smooth and drastically reduces the surface roughness.

Hardness is the resistance to surface indentation and scratching, which is a crucial clinical quality for maintaining surface smoothness and avoiding plaque accumulation, soft tissue irritation, wearing of the antagonist dentition, and resistance to discoloration [36]. In the current study for P specimens, the highest hardness mean value was in the VHN VP group, and the lowest was in the VHN HP group. The results agreed with the literature of Mei et al. [8], they demonstrated that 3D-printed zirconia, when constructed horizontally, frequently has pores concentrated close to its surface. These surface imperfections, being on the tensile side, would readily generate sites of stress concentration when subjected to a load.

Applying the glazed coating on the specimens resulted in an increase in microhardness values in all groups. As in Table VI, there were highly significant differences in microhardness between P and PG groups. In contrast, the study of Branco et al. [35] claimed that the hardness value of the glazed specimens was lower than that of unglazed ones because the underlying zirconia substrates have a hardness that is 3–3.5 times higher than the glaze layer. According to the author's knowledge, no other previous studies examined the effect of glazing on the surface roughness and microhardness of 3D-printed zirconia specimens in different printing orientations.

However, the limited number of specimens, materials, and equipment employed in this investigation, along with the small number of published articles addressing various orientations, reflect several limitations. Consequently, future research should examine additional 3D-printed zirconia brands, and incorporate other mechanical properties with various printing orientations. Moreover, evaluate specimens with color and translucency attributes.

CONCLUSION

The subsequent conclusions can be obtained through the recent investigation, taking into account its limitations:

1. The surface roughness and hardness values of AM zirconia were influenced by the construction angle.
2. The vertical orientation was the optimal printing direction compared to the horizontal and diagonal orientations in the surface roughness and microhardness values.
3. Glazing the 3D-printed zirconia specimens with diamond paste decreases the surface roughness of all zirconia groups except for the Diagonal orientation.
4. Glazing the 3D-printed zirconia specimens with diamond paste increased the microhardness values of all zirconia groups.

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None

Author's Contributions

NID: conceptualization. NID, ZNAW, SAN: data curation. NID, SAN: formal analysis. NID: funding acquisition. NID: investigation. NID, ZNAW: methodology. NID, ZNAW: project administration. NID: resources. NID: software. ZNAW: supervision. ZNAW, NID: validation. ZNAW, NID: visualization. NID, ZNAW: writing – original draft preparation. NID, ZNAW: writing – review & editing.

Conflict of Interest

The authors have no conflicts of interest to declare.

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Regulatory Statement

None.

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