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Surface roughness and translucency of glazed versus polished cubic ultra-translucent multi-layered zirconia: An *in-vitro* study

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Abstract

Statement of the problem: The final surface finish after sintering or after adjustment procedure of monolithic zirconia restorations is an important issue. Currently, there is lack of evidence based recommendations regarding the best surface finishing protocols for achieving optimal surface roughness and translucency of cubic multi layered ultra-translucent monolithic zirconia.
Aim: The purpose of this *in vitro* study was to assess the effect of surface finish protocols (glazing versus polishing) on the surface roughness and translucency of cubic ultra-translucent multi-layered zirconia.
Materials and Methods: CAD/CAM fabricated ultra-translucent multi-layer cubic zirconia cylinders were cut into disc-shaped samples and sintered to end up with (16) discs of uniform dimensions 11mm diameter and 0.6mm thickness. Disc samples were divided into two equal groups according to the surface finish protocols. Group G: glazing surface finish. Group P: polishing surface finish. Surface roughness (Ra) was assessed by a non-contact profilometer and translucent parameter (TP) was assessed by spectrophotometer.
Results: Surface roughness of studied samples of group 1 recorded (0.253±0.0009 µm) and (0.251±0.0024 µm) before and after glazing respectively. While for group 2 revealed (0.2547±0.0002 µm) and (0.2545±0.001 µm) respectively. While for translucent parameter of group 1 revealed (16.0553±2.659) and (15.3714±9.773) before and after glazing respectively. While for group 2 revealed (16.0553±2.659) and (14.242±8.325) respectively. There was a significant difference between both groups showing that glazing gave a smoother surface and more translucency than a polishing surface finish protocol.
Conclusions: Glazing of cubic ultra-translucent multi-layered zirconia is better than polished surface finishing protocol regarding surface roughness and translucency.

Keywords: Cubic zirconia, ultra-translucent zirconia, surface finishing protocol, polishing, multilayered zirconia, surface roughness and translucency

Introduction

Prosthetic dentistry is witnessing a trend toward monolithic ceramic restorations. This trend is being accompanied by the development of chair-side digital scanning and automated machining protocols for fabricating crowns and FDPs from ceramic blocks or discs [1].

Shade reproduction of natural teeth is an essential factor influencing the aesthetics of restorations. In addition one of the important factors to mimic naturality is translucency**.** The opaque appearance of earlier introduced zirconia negatively affected the esthetic outcomes. So seeking the excellent esthetic outcome a new generations of translucent zirconia have been made [2].

Translucent monolithic zirconia is one of the developed zirconia materials for anterior and posterior restorations. It combines the strength of zirconia with improved esthetics because of its higher translucency [3].

More recently the ultra-translucent multilayered zirconia has been introduced to the market. This material is intended to replicate the unique characteristics of teeth according to each anatomical region, keeping translucency at the incisal edge and a higher opacity at the cervical area [4].

Another factor influencing the appearance of the restoration is its surface roughness. To achieve smoothness surface polishing and glazing are routinely used. Both of these methods can alter the final view of the restorations and the practitioner should take into account the probability of color change after glazing or polishing [5, 6].

Our objective in this study is to evaluate different surface finishing protocols for cubic ultra-translucent multi layered zirconia to achieve optimal surface quality.

The first null hypothesis was; No difference would be encountered in the surface roughness of cubic multi layered ultra-translucent zirconia ceramics with both tested surface finish protocols (polished versus glazed). The second null hypothesis was; there would be no difference encountered in the translucency of cubic multi layered ultra-translucent zirconia ceramics with both tested surface finish protocols (polished versus glazed)

Materials and Methods

Ethical Approval

The study was approved by research ethics committee at Faculty of Dentistry Cairo University with an approval number of 19-12-3.

Sample size

Sample size was calculated depending on a continuous response variable from unmatched pairs in a previous study by [7]. According to this study, unmatched pairs were normally distributed with standard deviation (19.97). If the true difference in the mean response of unmatched pairs was (29.9), we need to study (8) pairs of sample to be able to reject the null hypothesis that this response difference is zero with probability (power 0.8 = 80%). The Type I error probability associated with this test of this null hypothesis is (0.05).

Sample preparation

A) CAD/CAM Fabrication

A cylinder of 14 mm length and 11 mm diameter was designed using 3D Builder software (Microsoft 3D Builder U.S.A) and saved as STL file, then the STL file was transferred to EXO-viewer (version 1.0.6136.31737, Germany)

The nesting procedures was done to include all of the four layers of the disc to mimic the situation of construction of conventional restoration [8].

A 5-Axis Dental Milling Machine (DWX-52D 5-Axis North America) was used for dry milling of cylinder made from Katana UTML Zirconia blank according to manufacturer instructions. Katana ultra- translucent multi-layer zirconia (UTML) 8 mol % Y_2O_3 zirconia polycrystal. Cylinders were milled to larger dimensions considering the amount of linear shrinkage that occurs after sintering, Figure (1) & (2) [8].

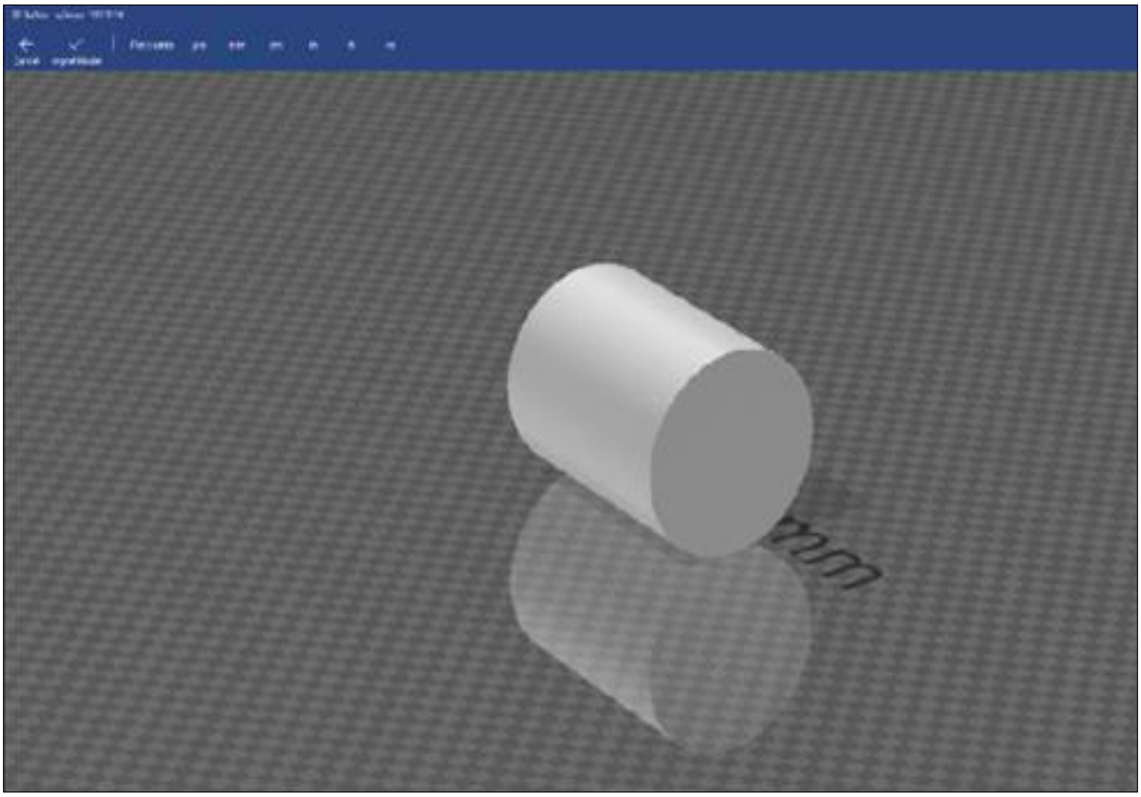


Fig 1: 3D design of cylinder ON 3D builder software

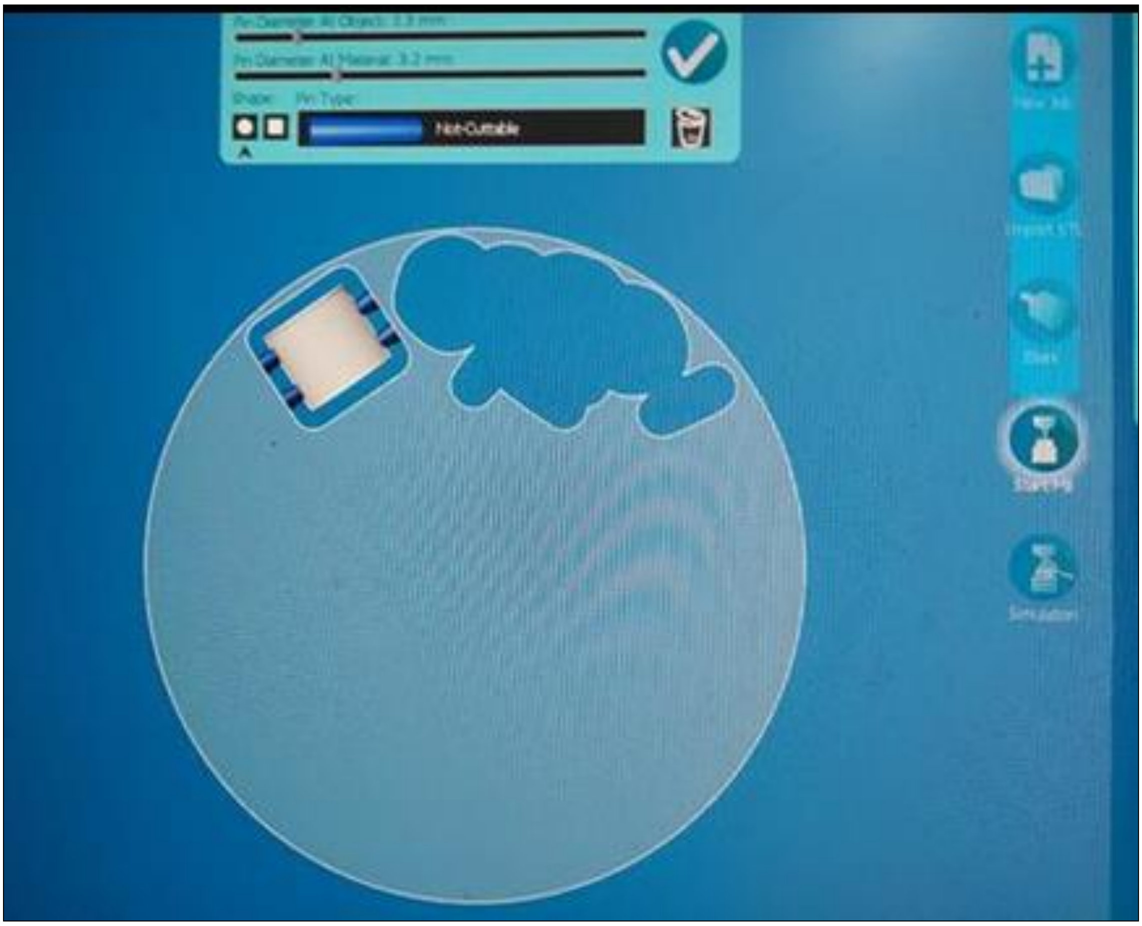


Fig 2: Cylinder nesting procedure inside zirconia disc on Exocad software

Sectioning of zirconia cylinder

Each cylinder was sectioned into 8 discs of 0.74 mm thickness using IsoMet4000 (Buehler, Lake Bluff, USA) at speed of 2,500 rpm, end up at 0.6 mm for a final thickness and the final diameter of the disc before sintering was 13.55 mm in diameter to end up with a final diameter of 11 mm. the isomet was calibrated to cut disc specimen of the specified thickness. It uses isocut wafering cubic boron nitride (CBN) blades with a thickness of 0.4mm, these blades work well for many tough materials giving shorter cut times. Figure (3) and (4). A total of 16 zirconia discs were produced. The measurements of width and thickness were then confirmed with a Digital caliper (Digital Vernier Caliper IP54, USA).

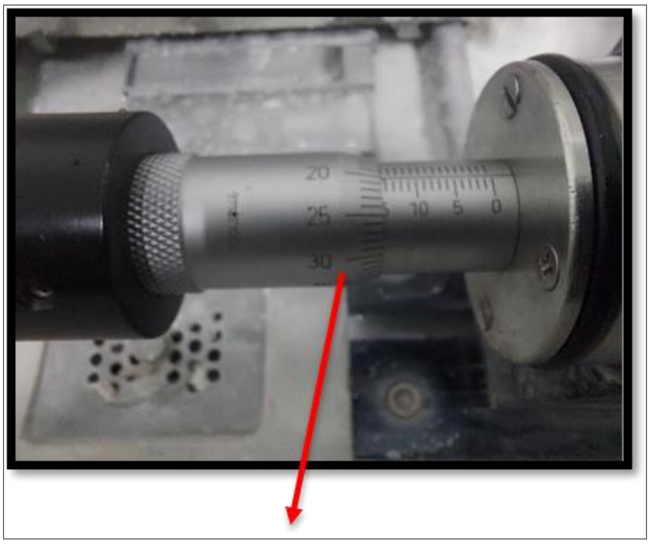


Fig 3: Internal caliper within Isomet

Fig 4: Sectioning of the zirconia cylinder with Isocut® Wafering Blades

Sintering procedures

All zirconia discs were subjected to an ultra-sonic cleaning using an Ultrasonic Cleaner device(Pt dent Ultrasonic Cleaner CD-4830 3L, techno flux, china) for 10 min in distilled water to remove any remnants from sectioning procedures followed by dry air streaming for 15 seconds.

Samples were divided into two groups of 8 discs each. Then was placed inside the sintering furnace (Wiessen Zirconia sintering furnace; Germany), samples were arranged over zirconia sintering beads, which reduce the atmosphere inside the sintering tray and prevent specimens from sticking or jamming during the sintering procedures. The sintering procedures was carried out following manufacture's guidelines for sintering.

The dimensions and thickness were re-checked after sintering procedures with a digital caliper to ensure that the amount of shrinkage that occurred after sintering is matched with that labeled on the Zirconia disc to end up with discs of 11mm in diameter and 0. 6 mm in thickness.

Randomization, allocation, concealment and implementation

A randomized sequence by which the samples in the study were allocated to the tested groups were generated using automated sequence generation (www.randomizer.org) to ensure the unpredictability of allocation sequence generated. Specimens were divided into two groups with a 1:1 allocation ratio, by randomly assigning numbers from 1 to 16. Sequentially numbered, opaque, sealed and stabled envelopes, specimens were numbered from 1-16 inside the envelopes while the allocation sequence was kept secret from the researchers. All steps of sample selection, randomization and preparation were assigned by the candidate under supervision.

Surface finishing of the samples

Glazing

For glazing, a considerable amount of ready-made paste of clear glaze (Kuraray Noritake Dental Inc (Japan)) was applied evenly covering the entire single surface of each disc using a clean brush .The discs were then placed on a honeycomb firing tray for glazing in porcelain firing furnace (Programat P200 - Dental Porcelain Furnace),With power firing cycle according to manufacturer's instructions.

Polishing

Disc polishing was carried out using a dental surveyor '' surveyor II (Surveyor II, Saeshin Precision Co., Ltd., Korea) to assure standardization of the procedures. A Teflon mold was milled with 11mm hole to fit the disc. Sticky wax was used to fix disc into the mold during polishing [9].

For standardization of both direction and pressure, the Teflon mold was fixed in the surveyor using a specially designed gypsum cast model, Figure (5).

The selected polishing instrument was mounted in a straight low-speed hand piece of the micro-motor (Strong Micro Motor 207, Kore) that was fixed to the upper member of the surveyor in such a way that the strokes of the stone attached to the hand piece are applied parallel to the long axis of the disc.

Using Diacera Polishing (Diacera polishing kit for zirconia EVE Ernst Vetter GmbH) technical set the discs were polished with green pre- polishing rubber at speed of 10.000min-1 then high gloss pink polishing rubber were used at a speed of 6.000 min-1 according to manufacturer's instructions to produce high surface luster in time of 60 seconds for each step. All polishing steps were done in a single direction and with a steady pressure. Finally for extra surface finish a goat wheel polishing tip was used [10].

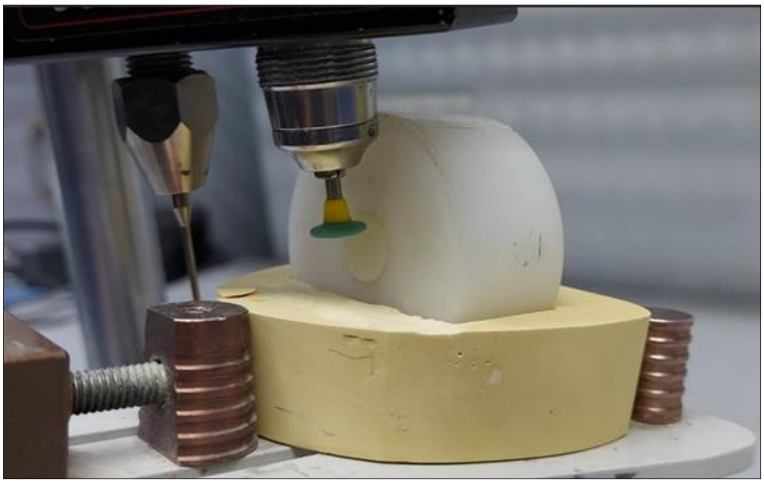


Fig 5: Cast model holding the Teflon mold parallel to the straight hand piece

Testing

Surface roughness examination

Surface roughness measurements for zirconia discs were performed twice: after sintering to give baseline readings and after application of the surface finishing protocols (glazing vs. polishing).

Measurements were done using the optical profilometry (Optical profilimeter U500X, Digital Microscope) Guangdong, China). Discs were photographed using USB Digital microscope with a built-in camera (Digital Microscope) connected with an IBM compatible personal computer using a fixed magnification of 120X.

Images were cropped to 350×400 pixels to specify/standardize area of roughness measurement and analyzed using WSxM software (Version 5) (WSxM software, Nanotec Electronica S.L., Madrid, Spain).

WSxM software was used to calculate average of heights (Ra) expressed in (µm), which can be assumed as a reliable indices of surface roughness.

The percentage of change was calculated according to the following formula

$$\text{Percent of change in (Ra)} = \frac{\text{(Ra) after surface finish} - \text{(Ra) before surface finish} \times 100}{\text{(Ra) before surface finish}}$$

Moreover, qualitative evaluation of the surface morphology of the discs was done using scanning electron microscope (Quanta™ 250 FEG, FEI Company, United states).

Translucency measurement

The translucency measurements were performed for each specimen over a white backing with a specified parameters (CIE L*= 88.81, a*= -4.98, b*= 6.09) and black backing with the specified parameters of (CIE L*= 7.61, a*= 0.45, b*= 2.42) relative to the CIE standard illuminant D65.

The translucency parameters (TP) values were obtained by calculating the color difference of the specimens over black and white backgrounds by using the following equation:

TP= [(Lb-Lw) ^{2}+ (ab-aw) ^{2}+ (bb-bw) ^{2}] ^{½} [11].

Then initial and final data were collected before and after applying both tested surface protocol glazing and polishing and percentage of change was calculated according to the following formula

$$\text{Percent change of (TP)} = \frac{\text{(TP) after surface finishing} - \text{(TP) before surface finishing}}{\text{(TP) Before surface finishing}} \times 100$$

Results

A descriptive study performed on surface roughness of studied samples before and after glazing. Mean ± standard deviation revealed (0.253±0.0009 µm) and (0.251±0.0024 µm) before and after glazing respectively. While after polishing, mean ± standard deviation revealed (0.2547±0.0002 µm) and (0.2545±0.001 µm) respectively, as listed in table (1) and showed in figure (6).

Using paired t test for dependent variables, it revealed that there was insignificant decrease in surface roughness by (-0.7668%) and (-0.0825 µm) respectively for glazing and polishing, as P-value > 0.05. Finally, performing independent t test for significance estimation between both independent groups revealed that there was significant difference between both groups as P-value< 0.05, as listed in table (1).

While for translucency evaluation, a descriptive study performed on studied samples before and after glazing. Mean ± standard deviation revealed (16.0553±2.659) and (15.3714±9.773) before and after glazing respectively. While after polishing, mean ± standard deviation revealed (16.0553±2.659) and (14.242±8.325) respectively, as listed in table (2) and showed in Figure (7).

Using paired t test for dependent variables, it revealed that there was insignificant decrease in translucency by (-0.04259%) and (-0.11291) respectively for glazing and polishing, as P-value > 0.05. Finally, performing independent t test for significance estimation between both independent groups revealed that there was significant difference between both groups as P-value< 0.05, as listed in table (1).

Table 1: Descriptive Study and Comparative Study of Effect of Glazing and Polishing on Samples Surface Roughness:

	Before	After	% change	P-value (Paired t-test)
	M±SD	M±SD		
Group I (Glazed)	0.253±0.0009 µm	0.251±0.0024 µm	-0.7668	0.1078 (ns)
Group II (Polished)	0.2547±0.0002 µm	0.2545±0.001 µm	-0.0825	0.5579 (ns)
P-value (Independent t-test)			<0.0001*	

M; Mean, SD; Standard Deviation, %; Percentage of Change, P; Probability Level
Ns; insignificant Difference
*Significant Difference

Table 2: Descriptive Study and Comparative Study of Effect of Glazing and Polishing on Samples Translucency:

	Before	After	% change	P-value (Paired t-test)
	M±SD	M±SD		
Group I (Glazed)	16.0553±2.659	15.3714±9.773	-0.04259	0.8333 (ns)
Group II (Polished)	16.0553±2.659	14.242±8.325	-0.11291	0.5200 (ns)
P-value (Independent t-test)			<0.0001*	

M; Mean, SD; Standard Deviation, %; Percentage of Change, P; Probability Level
Ns; insignificant Difference
*Significant Difference

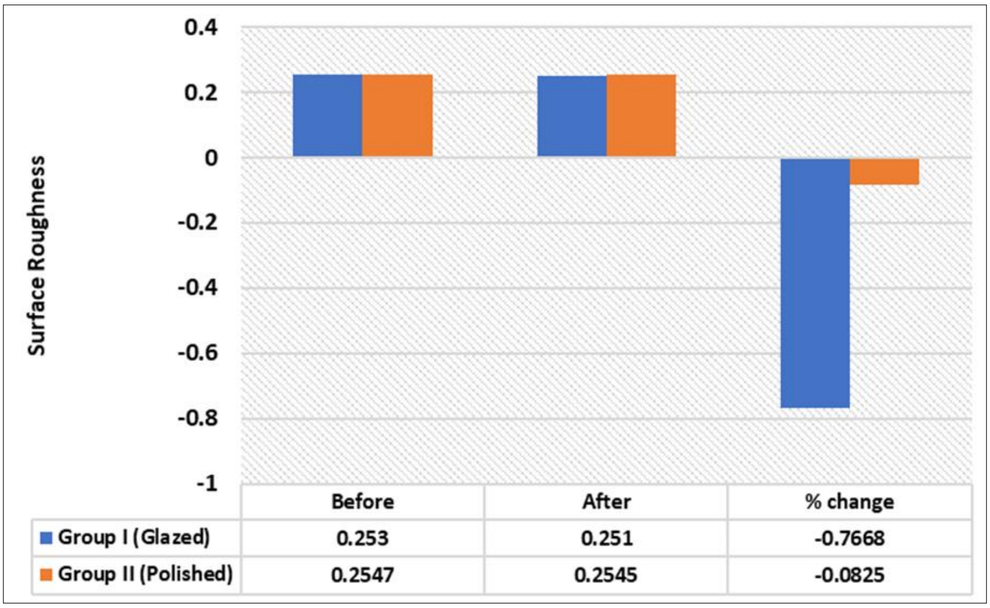


Fig 6: Bar Chart revealing Effect of Glazing and Polishing on Samples Surface Roughness

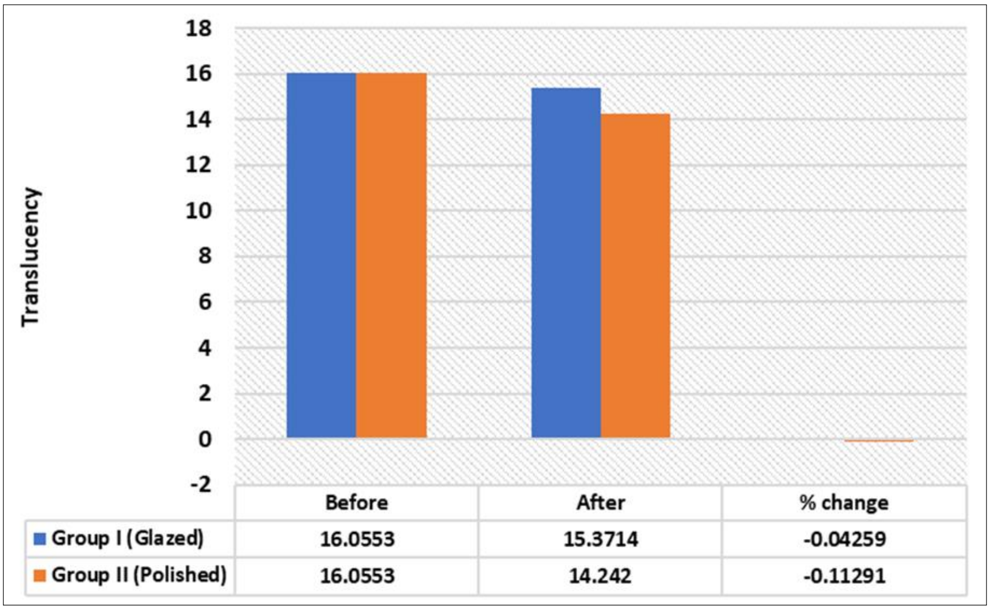
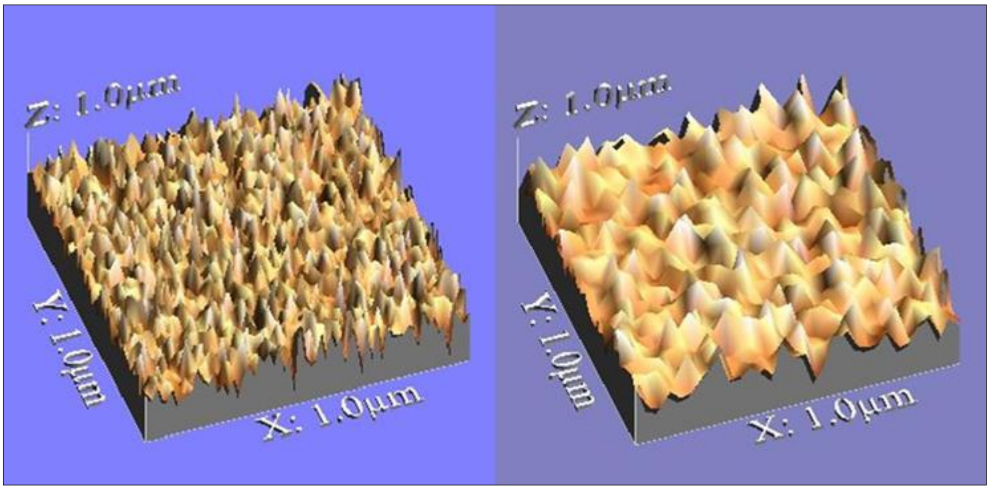


Fig 7: Bar Chart revealing Effect of Glazing and Polishing on Samples Translucency

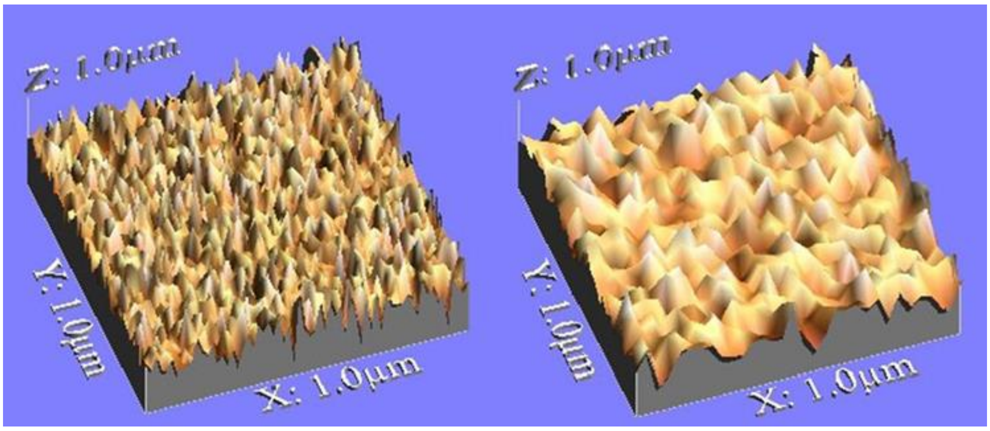
Results of the optical profilometer testing of the surface roughness:

The WsxM images demonstrated a 3D images of the surface roughness change pre and post surface finish where the changes were seen in the difference in heights of the peaks and depth of the valleys across the surface. In both tested surface finishing protocols (glazing & polishing), discs exposed to polishing surface finish gave the highest roughness followed by discs exposed for glazing surface finish Figure (8, 9, 10).



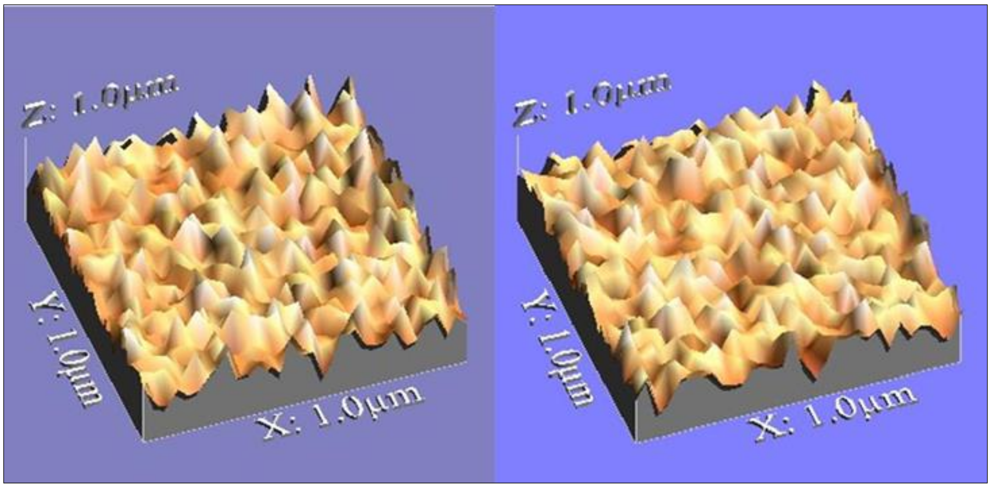
A) Before polishing B) After polishing

Fig 8: WsxM images showing surface roughness of polished samples a) before polishing and b) after polishing



A) Before glazing B) after glazing

Fig 9: WsxM images showing surface roughness of glazed samples: a) before glazing and b) after glazing

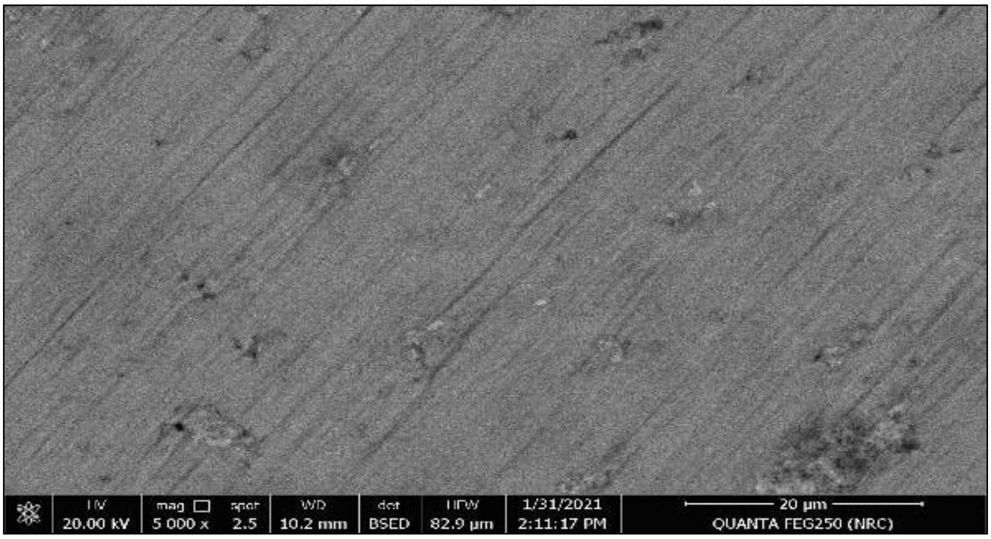


A) After polishing B) After glazing

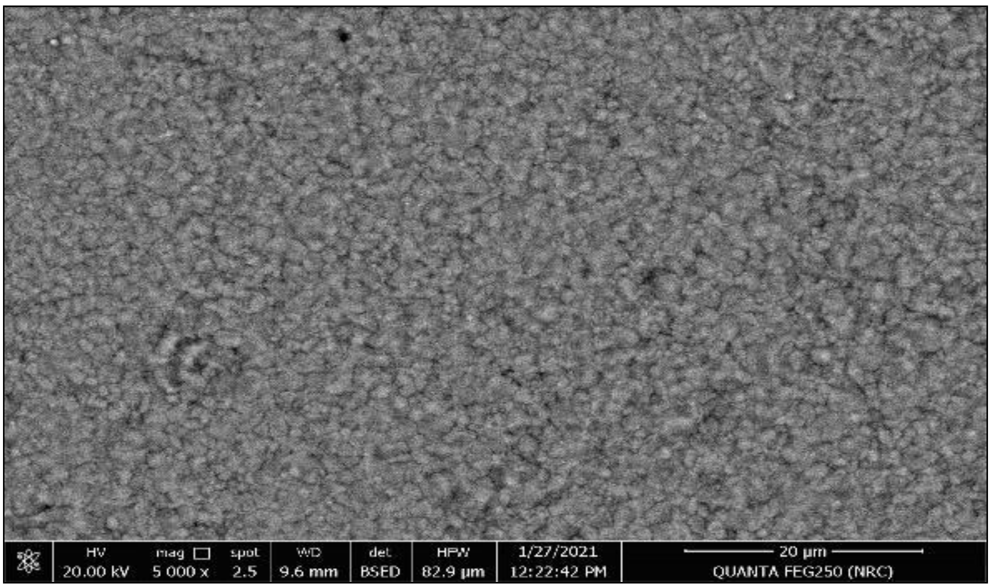
Fig 10: WsxM images showing surface roughness of zirconia samples e) after polishing f) after glazing.

Results of the SEM examination

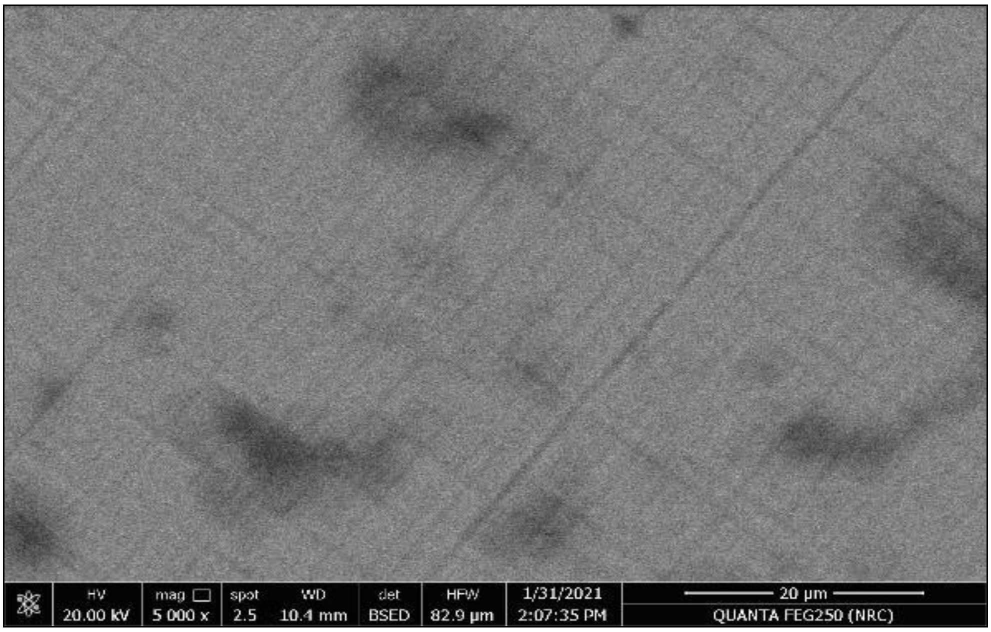
The specimen was subjected to SEM before any surface treatment for compassion as shown in Figure (11a). The SEM image of glazed sample showed homogenous surface morphology due to the glaze layer covering the surface entirely which made the crystals hardly distinguished as seen in Figure (11b). While the samples exposed to polishing surface finishing showed obvious surface change seen as numerously scattered patches &small pores that might indicate high surface roughness as shown in Figure (11c).



a) Specimen before subjecting it to surface finishing protocol



b) Specimen after being subjected to glazing finishing protocol



c) Specimen after being subjected to polishing finishing protocol

Fig 11: Scanning electron microscope photo showing the surface of cubic ultra-translucent zirconia a) before subjecting it to any surface finishing immediately after sintering, b) after subjecting it to glazing and c) after subjecting it to surface polishing.

Discussion

In recent years the use of yttrium stabilized zirconia (YSZ) ceramics to manufacture full contour monolithic restorations as single-unit fixed dental prostheses (FDPs) has been considered. One of the promising types of monolithic ZR is the ultra-translucent multi-layer zirconia ceramic [12].

The composition of this material would offer the excellent aesthetic performance of glass ceramics combined with higher translucency in comparison with conventional zirconia. Also, this material would provide higher flexural strength and fracture toughness when compared to lithium disilicate glass ceramics, as reported by Burgess, (2018) [13], and Hynková *et al*., (2021) [14]. Additionally, it is important to highlight that increased resistance to low temperature degradation is suggested with this type of zirconia [15, 1].

The thickness of the milled specimens was 0.6 mm. The new translucency zirconia exhibits favorable mechanical properties and esthetic performance at 0, 5-1mm which enables the dentist to be more conservative during tooth preparation and give maximum esthetics at the same time [3]. The diameter of the zirconia discs was chosen to be 11 mm to be easily handled during the glazing and polishing steps.

CAD/CAM production of zirconia discs was considered designing was done using 3D builder software to ensure accuracy. And milling was done using a five axis milling machine. This method shows the advantages of being an efficient, time-saving, errorless, and standardized method to produce an accurate specimen. Dry milling was carried out to avoid softening of non-sintered zirconia that may be caused by wet milling, as reported by, Alghazzawi, (2016) [16], and Jum'ah *et al*., (2020) [17]. Finally, cylinders were sectioned using Isomet 4000 at speed of 2,500 rpm under water coolant to end up with 16 discs and sintered according to manufacture instructions.

In the current study two surface finish protocols were tested (Glazing vs. polishing). Polishing has been reported to be a successful technique in attaining high surface finish and reduce surface roughness with results comparable to glazing procedures. Glazing is the most common method used to smoothen rough surfaces. The glaze layer fills the micro-cracks and the porosities created on the surface. In addition, several studies showed that glazing increases the strength of the material [3].

Testing of surface roughness was carried out in the present study both quantitatively using optical profilometer and qualitatively using scanning electron microscope.

The Ra value as a roughness parameter has been used in the present investigation, because this is the most commonly used value for roughness determination and it, therefore, allows easier comparability to other studies, easy to define, easy to measure, and gives a good general discerption of height variations [18].

Translucency was assessed in the present study before and after surface finishing protocols using the translucency parameter (TP) as it expresses the ability of a dental material to hide differences in the background and is calculated as a color difference of a material against a black and white background [19, 12].

The first null hypothesis stating that no difference would be encountered in the surface roughness of cubic zirconia ceramics with both tested surface finish protocols (glazed versus polished) was rejected as the current study revealed that there was a significant difference between both tested groups.

Regarding the results of the surface roughness concerning the glazing surface finish protocol, it was noticed that the reduction in surface roughness before and after glazing was insignificantly different and were within the accepted range of surface roughness of the restoration inside the oral cavity. Surface roughness less than or equal to ≤ 0.2 µm provides minimal plaque accumulation and a comfortable tactile sensation [18].

These results coincide with Khayat *et al*., (2018) [20] who showed that the roughness of translucent zirconia was not affected by glazing procedures. On the contrary, Al Hamad *et al*., (2019) [21] found that there were significant differences between the roughness of the unglazed and glazed surface of both opaque &translucent monolithic zirconia.

Also, our results are in disagreement with Manziuc *et al*., (2019) [3] who measured Ra of two types of monolithic zirconia (6.5%-8%) of yttria and they found that glazing decreased the surface roughness of monolithic zirconia.

Both previously mentioned studies show different results from the current study this could be attributed to their using of different type of zirconia, different glazing material and addition of a separate polishing step before glazing was performed, cold have affected the surface roughness.

With regards to the polishing surface finish protocol, it was noticed that the reduction in surface roughness before and after polishing was insignificant.

This finding is in agreement with Al Hamad *et al*., (2019) [21] who reported that polishing showed no effect on roughness of zirconia ceramics.Also, our results coincide with the finding of Jum'ah *et al*., (2020) [17] who reported that two-step polishing protocols for 5Y-ZP and 8Y-ZP using the Diacera finishing kit, showed no statistical significance in roughness reduction. On the contrary,

There was a significant difference between both groups showing that glazing gave a smoother surface than a polishing surface finish protocol. As the polishing finishing protocols leave surface scratches on the discs, while the glazing surface finish seals the porosities and surface flaws which gives a smoother surface and these results were supported by the scans done using the scanning electron microscope.

These findings could be attributed to the polishing procedures being done after sintering only and not in the white stage (pre-sintered stage). Also, the type of polishing kit used for polishing specimens is a coarse finish kit, not a fine polishing kit. Using of fine polishing kit might have influenced the final results. This was recently explained by Poosanthanasarn *et al*., (2022) [22] they used both coarse and fine polishing kits, and this caused a dramatic change in the surface roughness of translucent zirconia.

These results are in agreement with Kim *et al*., (2016) [23] who stated that Glazing produces the smoothest surface regardless of the number of colorings in comparison to the polishing of monolithic zirconia.

Moreover, Jum'ah *et al*., (2020) [17] reported that glazing exhibited significantly lower surface roughness when compared to all polishing protocols. Furthermore, ultra-translucent cubic zirconia demonstrated inferior polishability compared to 3Y-TZP.

Additionally, Teja *et al*., (2021) [24] agreed with our study results when they used cubic zirconia zolid HT with different surface finishing, they reported that the glazed specimen showed the least surface roughness in comparison to polishing.

On the other hand, the results of the present study are in disagreement with Khayat *et al*., (2018) [20], who found that polishing surface finish protocol produced a comparable surface roughness to glazing surface finishing protocol.

Giti *et al*., (2020) [25] also disagreed with our results as they reported that the surface roughness of glazed specimens was significantly higher than polished and unpolished specimens. The difference may be due to the use of a different brand of finishing kits (KAVO Germany) or the use of different cubic zirconia brands (Zircostar, Kerox) which have 5.7-9.8 wt% of yttria.

Furthermore Toma *et al*., (2022) [26] stated that surface treatment (glazing or polishing) for three types of translucent multilayer zirconia has a significant impact on surface roughness and micro hardness. The glazed samples were found with higher surface roughness and lower microhardness compared to the polished ones.

The findings of the present study could also be attributed to that the use of zirconia grinding burs that may result in deep surface flaws that can be very difficult to polish especially in 5Y-ZP/8Y-ZP materials. The 4-step polishing protocol including both coarse and fine polishing rubbers was associated with the finest surface finish and least residual surface flaws. The only polishing protocol that resulted in Ra comparable to glazing was the 4-step protocol when applied to 3Y-TZP which also highlights that the polishing protocol should have multiple steps to reach the level of smoothness of glazing procedures as reported by, Jum'ah *et al*., (2020) [17], Shin & Lee, (2021) [10] reported that finely polished zirconia showed better surface roughness than glazed ones when using 3-step polishing burs.

Regarding the results of the effect of surface finish protocols glazing versus polishing on the translucency of Cubic ultra-translucent zirconia ceramic, there was a significant difference between both tested groups where glazing gives a higher translucent restoration than polishing.

These findings were in line with Manziuc, *et al* (2019) [3] who concluded that translucency changed after glazing for the translucent cubic zirconia tested. These findings were also in agreement with Saker & Özcan, (2021) [12] who found that glazing always gives a higher translucency than polishing groups even after aging which is in agreement with the present finding.

These findings were against Kim *et al*., (2016) [23] who investigated the effects of glazing and polishing on the translucency, and opalescence of dental monolithic zirconia ceramics, they summarized that surface treatment did not significantly affect the translucency of monolithic zirconia ceramics in most groups.

Finally, from the point of view of the authors, the surface roughness and translucency properties are material dependent. moreover, the most suitable polishing protocol is not yet defined. However, care must be taken to include both coarse and fine polishing steps to reach the aimed smoothness.

Conclusions

Within the limitations of this study, the following could be concluded

1. The roughness of cubic ultra-translucent multilayered zirconia ceramic was reduced by glazing surface finish protocols in comparison to polishing.
2. The translucency of cubic ultra-translucent multilayered zirconia ceramic was improved by the glazing surface finish protocol.
3. Surface roughness produced by the surface finishing protocols is a crucial factor in determining translucency of the cubic ultra-translucent multilayered zirconia ceramic.

Recommendations

- Further studies are recommended to test the effect of different finishing protocols on the optical properties of cubic ultra-translucent zirconia ceramic restoration after thermo-mechanical aging.
- Further investigations are recommended to test the effect of different finishing protocols on the mechanical properties of cubic ultra-translucent zirconia ceramic restoration.
- Further clinical studies are required to assess the efficiency of different surface finishing protocols with cubic ultra-translucent zirconia ceramic restoration.

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Conflict of Interest
The authors declare that they have no conflict of interest regarding the publication of this paper.

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