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ASSESSMENT OF ULTRA VERSUS HIGH TRANSLUCENT ZIRCONIA: SURFACE ROUGHNESS AND COLOR STABILITY (IN- VITRO STUDY)

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ABSTRACT

Aim: To assess the effect of accelerated aging on the surface roughness and color stability of ultra-translucent and high-translucent zirconia.

Materials and methods: A total of sixteen discs (11mm diameter and 0.6mm thickness) were sliced from two materials; ultra (UTML) and high (HTML) translucent multilayered zirconia; Group UT and HT respectively. All samples were polished and subjected to accelerated aging using autoclave for 5-hours to simulate 15-years clinical service. Surface roughness was assessed using digital microscope and SEM. Color stability was assessed using laboratory spectrophotometer. After testing for normality, collected data revealed parametric distribution and were expressed as mean and SD. Data were statistically analyzed using Paired t-test and independent t-test at a level of significance (*P*£0.05).

Results: Before aging, the mean surface roughness was (0.2508µm ±0.00152) and (0.25017µm ±0.00155) for Groups UT and HT respectively. After aging the mean surface roughness was (0.2533mm ±0.0019) and (0.2536mm ±0.0022) for Groups UT and HT respectively and DE were (3.05 ± 0.31) and (3.71 ± 0.42) respectively. Paired-t test revealed that surface roughness increased significantly. After aging, however independent t-test revealed that there was in-significant difference between both groups regarding surface roughness, while significant difference regarding color stability was found.

Conclusion: It was concluded that accelerated aging significantly affects the surface roughness and color stability of ultra and high translucent zirconia.

KEYWORDS: Ceramics; Zirconia; Accelerated aging; Surface roughness; Color stability; Scanning electron microscope.

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INTRODUCTION

The increased demand for esthetics has led to using all-ceramic approaches as alternatives to metal-ceramic restorations [1]. The main advantages of all ceramic restorations were their high chemical stability, biocompatibility, corrosion resistance, high hardness and wear resistance and most important their excellent esthetics and durability [2].

Conventional zirconia (first generation) known as tetragonal partially stabilized zirconia (3Y-TZP) offered superior mechanical properties, but its high opacity and intense whiteness represented an esthetic limitation [3]. This led to the introduction of a second generation of zirconia, with the number and size of Al_2O_3 grains being reduced and relocated in the zirconia framework (3). The repositioning of Al_2O_3 grains, whose refraction index varies greatly from that of zirconia grains, on the grain boundaries of zirconia, allowed higher light transmittance with simultaneous long-term stability and high strength [3].

A third-generation of zirconia was further introduced, which was not only metastable in the tetragonal phase, but also contained a cubic phase proportion of up to 53%. It was described as fully stabilized zirconia with a mixed cubic/tetragonal structure [3]. Cubic portions were achieved through adding higher amount (9.3 wt%-5 mol%) of yttrium oxide [4,5].

The cubic crystals have a higher volume compared to the tetragonal ones; causing the light to scatter less strongly at the grain boundaries and residual porosities, making the material more translucent. Furthermore, the cubic crystal structures are more isotropic than the tetragonal structures, meaning that incident light is emitted more evenly in all spatial directions, which significantly influence translucency [4]. Cubic zirconia has lower flexural strength (600-800 MPa), however, compared to layered zirconia, their flexural strength is considered higher. In addition, cubic zirconia does not have the capability to perform a tetragonal to monoclinic phase transformation as tetragonal zirconia does, thus, eliminated the unique transformation toughening that gives zirconia its toughness and resistance to fracture [6,7].

Although, monolithic zirconia overcame chipping fractures of veneered restorations, they were monochromatic. In recent years, multilayered zirconia systems were introduced to mimic the shade gradient of natural teeth [8].

Zirconia restorations showed high success due to their good chemical stability, biocompatibility, high fracture toughness and wear resistance. However, when in contact with water or water vapor, zirconia suffered from a slow, spontaneous and progressive phase transformation of the metastable tetragonal phase to the monoclinic phase known as aging or low temperature degradation (LTD) phenomenon [9].

To understand the LTD phenomenon in in-vitro studies, a laboratory simulation (artificial aging) is usually performed to mimic the clinical intra-oral conditions. Such procedure is possible through thermocycling, autoclave accelerated aging and weathering process [10]. Autoclave accelerated aging is a useful method; using steam autoclave treatments at increased temperatures (120°C to 140°C) under pressure to allow LTD in the presence of water, where the tetragonal/monoclinic phase transformation is thermally activated and accelerated. One hour of artificial aging treatment in an autoclave at 134°C under 0.2MPa pressure is equivalent to 3 to 4-years in vivo at 37°C [10].

Surface roughness is considered an important factor affecting restoration's long-term survival; jeopardizing the biomechanical and esthetic values and promoting the effect of aging in zirconia[10]. Roughness is also a good predictor of the restoration's mechanical performance, since surface irregularities may form initiation sites for cracks or corrosion with subsequent failure [10,11]. Restoration surface is considered smooth when the surface roughness is below 0.2µm, however, more than 0.5µm is set for the roughness threshold detectable by the tongue [5].

Color stability is also crucial [12]. Instrumental analysis using spectrophotometers can be used to capture and analyze color. Laboratory spectrophotometer offered high accuracy in detecting color parameters, providing objective numerical values, while eliminating the effect of human factors. Compared to conventional visual assessment, laboratory spectrophotometers offered a 33% increase in accuracy [13].

Ultra and High translucent multilayered zirconia, being newly introduced, their behavior after accelerated aging is not fully reported.

The aim of the present study was to assess the effect of accelerated aging on surface roughness and color stability of polished Ultra and High translucent multilayered zirconia. The null hypothesis was that accelerated aging would have no effect on the surface roughness and color stability of polished Ultra-translucent multilayered zirconia compared to polished High-translucent multilayered zirconia.

MATERIAL AND METHODS

A total of sixteen-disc samples (n=8; diameter=11mm, thickness=0.6mm) were prepared from Ultra-translucent multilayered katana zirconia (UTML; Group UT) and High-translucent multilayered katana zirconia (HTML; Group HT) blanks (Table 1). The number of samples were determine based on power analysis performed prior to conducting the study.

Disc specimens' construction:

Uniform cylinders were milled from UTML AND HTML blanks (Kuraray Noritake Dental Inc., Japan). The obtained cylinders aided in standardizing the diameter of the specimens to be tested in both groups. The cylinders were designed using a CAD software (3D Builder, v.2019), sent as STL file to CAM software (DGSHAPE MillBox Edition, v.3.7.3) to allow for proper nesting within the blanks to include all of the four layers of the disc (dentin layer, first transitional layer, second transitional layer, and enamel layer). Each cylinder was milled from its corresponding blank, using 5-axis milling machine (DGSHAPE DWX-52D, Japan).

Cylinders were sliced using linear precision saw (Buehler IsoMet 4000 Precision Cutter, Illinois, USA) at speed of 2500rpm, which offered high cutting efficiency and eliminated surface damage[5] under water coolant to reduce heat generation. Uniform discs (0.72mm thickness) were 25% larger than the specimens desired final size to compensate for sintering shrinkage.

TABLE (1) The materials tested in the present study

Commercial Names	Chemical Composition	Physical Properties	Mechanical Properties	Manufacturer	LOT No.
Ultra translucent multilayered Katana Zirconia disc (A2, T14)	-75 wt% c-ZrO2 - yttrium (Y2O3) content: 9.55%Y - no detectible Al	- Coefficient of thermal expansion (25-500°c): 9.7 ± 0.2 10-6 K-1 -Transmittance: 43%	Flexure strength: 557Mpa	Kuraray Noritake Dental Inc., Japan	DMZMG
High translucent multilayered katana zirconia discs (A2, T14)	-<50 wt% c-ZrO2 -yttrium (Y2O3) content: 5.2 %Y -0.06 wt% Al	- Coefficient of thermal expansion (25-500°c): 9.9x10-6 /K -Transmittance: 31%	Flexure strength: 1125Mpa		ECLCN

All disc specimens were checked carefully using magnifying lens for any surface defects, in addition to a precise digital caliper (Digital Vernier Caliper IP54, USA) to verify the disc thickness. All disc specimens were cleaned using ultrasonic cleaner (PT dent Ultrasonic Cleaner CD-4830 3L, Techno flux, China) in alcohol solution for 10-minutes and air dried to eliminate of any sectioning residues [10]. Specimens were then sintered (Wiessen Zirconia sintering furnace, Germany) according to the manufacturer instructions of each material. After sintering, specimens were rechecked with the digital caliper to verify the final thicknesses (0.6mm) and diameter (11mm).

Polishing of the specimens

All specimens were polished using a polishing kit for zirconia (Diasynt plus & Diacera zirkonoxid zirconia Eve, Germany). To standardize such procedure, a special custom-made Teflon specimen holder was fabricated to hold the specimen during the finishing and polishing procedures, which was accomplished by a surveyor (Surveyor-II, Saeshin Precision., Ltd., Korea) holding the polishing tools (Figure 1) During the polishing procedure the selected instrument was mounted on a straight hand-piece, connected to low-speed micro-motor (Strong Micro Motor 207, Korea) that was fixed to the upper member of the surveyor in such a way that the stone attached to the hand piece would be parallel to the long axis of the specimen. Polishing was performed according to the manufacturer instructions, using green medium DIACERA rubber polisher at speed of 10.000rpm for pre-polishing, then, orange fine DIACERA rubber polishers at speed of 6.000rpm for high-shine polishing for 60-seconds for each step, in one direction (up to down) by manually moving the surveyor in the specified direction. Polishing of all samples was performed by single operator to ensure standardization.

After polishing completion, each specimen was randomly placed in a small numbered sealed plastic bag. The plastic bag protected the specimens from any accidental scratches. The numbers used helped in blinding the assessors and statistician to the tested groups and in proper results documentation. Randomization helped reduce bias and ensured results reliability [14].

P.re-aging surface roughness assessment: Specimens were tested for surface roughness using USB Digital microscope (U500X, Guangdong, China) at magnification of 120X. The captured images were analyzed using WSxM software (Nanotec Electronica S.L., Madrid, Spain)[15,16] to calculate the average heights (Ra; arithmetic average surface roughness) expressed in µm. Scanning electron microscope (Quanta FEG-250 SEM FEI Company, Eindhoven, Netherlands) was used for surface roughness assessment at 500X and 10000X magnification.

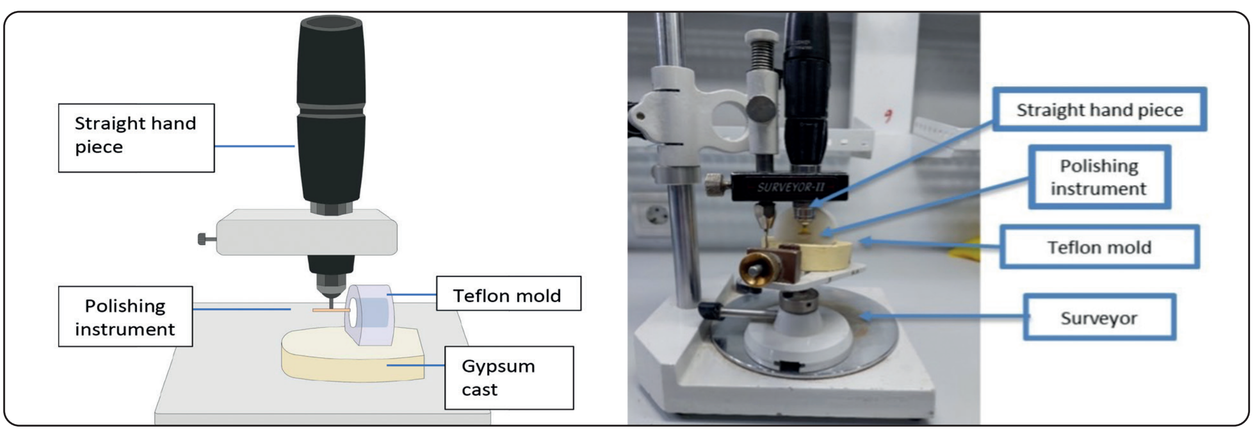

Fig. (1) Custom made apparatus used for polishing specimens

Pre-aging color assessment: The specimens color was measured using a reflective spectrophotometer (Agilent Cary 5000 UV-Vis-NIR spectrophotometer, Australia). This served as a baseline reference. The measurement was performed at the center of each specimen over a black background (CIE L*=7.61, a*= 0.45, b*=2.42) relative to the CIE standard illuminant D65. The spectrophotometer was calibrated before each measurement. Three measurements were taken for each specimen and the average was recorded.

Accelerated Aging

All specimens were subjected to artificial accelerated aging using autoclave (HaTmed Q70B, Germany) at temperature of 134˚C and 2-bar (0.2Mpa) pressure for 5-hours (10-cycles) in accordance to ISO standards 13356 to simulate oral conditions of 15-years [11]. Each specimen was placed in a sterilization pack labeled with its specified code and placed in the autoclave widely separated to avoid specimens overlapping. After aging, all specimens were air-dried for 24-hours before testing [5].

Post aging testing procedures

Surface roughness was evaluated after aging as previously explained using the same devices. Color was also assessed as described previously and the color change (ΔE) was calculated [10], **where:**

$\Delta E = [(L^* \text{ after aging} - L^* \text{ baseline})^2 + (a^* \text{ after aging} - a^* \text{ baseline})^2 + (b^* \text{ after aging} - b^* \text{ baseline})^2]^{1/2}$

Where L* represented lightness-darkness, while a* and b* represented redness-greenness and yellowness-blueness, respectively

Statistical Analysis

The collected data for all outcomes were quantitative in nature. They were analyzed by an expert statistician, who was blinded to the tested materials. The statistical analysis was performed using Statistical Package for Social Science (SPSS)® Ver.24 (IBM Product, USA), Minitab® statistical software Ver.16 (Minitab LLC, USA) and Microsoft Excel® 2016 (Microsoft Cooperation, USA).

Data were first explored for normality using Shapiro-Wilk test and Kolmogorov-Smirnov test. Surface roughness and color stability data were found to be normally distributed and were presented as mean and standard deviation (SD).

Paired t-test was used to compare surface roughness between dependent values obtained before and aging within each group. Independent t-test was used to compare between tested groups in regard to surface roughness and color stability, where the level of significance was set to $P \leq 0.05$. Further analysis was performed to determine the percent of change before and after aging in both groups regarding surface roughness and color stability according to the following formula:

$$\text{percent change} = \frac{\text{final} - \text{initial}}{|\text{ initial }|} \times 100$$

RESULTS

Surface roughness results

Quantitative Data: The surface roughness of both groups was significantly higher after aging. Comparing the mean values revealed an insignificant difference between both groups before and after aging, where HT Group was higher than UT Group before and after aging. However, comparing the percentage of change of surface roughness showed a significance difference between both groups, with HT Group was higher than UT Group. (Table 2)

Roughness pattern: The obtained 3D images revealed that the surface roughness pattern before aging in both Groups (UT and HT) was comparable with relatively uniform distribution of shallow valleys and broad peaks across the surface. However, after aging both groups showed an increase in the

TABLE (2) Results of surface roughness in both groups before and after aging

Results of surface roughness in both groups before and after aging					
Surface Roughness	Group UT		Group HT		P-value
	M	SD	**M**	SD	
Before Aging	0.2508 mm [a]	0.00152	0.25017 mm [a]	0.0015	**0.3772**
After Aging	0.2533 mm [b]	0.0019	0.2536 mm [b]	0.0002	**0.7926**
P-value	**0.001***		**0.024***		---------------
% Of change	0.95%	0.0061	1.352%	0.0041	**<0.0001***

*M: Mean, SD: Standard Deviation, P: Probability Level which is significant at P ≤ 0.05, *: Significant Difference*

TABLE (3) Results of color stability in both groups before and after aging

Results of color stability (ΔE) in both groups					
ΔE	**Group UT**		**Group HT**		
	3.05	0.31	3.71	0.42	**0.003***

*M: Mean, SD: Standard Deviation, P: Probability Level which is significant at P ≤ 0.05, *: Significant Differenc*

surface roughness manifested by increasing the peaks heights and valleys depth across the surface, with the highest roughness seen in aged HT Group. (Figure 2)

Scanning Electron Microscope (SEM): SEM images obtained at 500X magnification for both groups (UT and HT) before aging were relatively smooth, with Group UT showing minor shallow linear defects. After aging, SEM images showed increased roughness for both groups with different patterns, where Group UT showed irregular small uniform defects spread across the surface, whereas, Group HT showed numerous linear grooves overlapping each other in addition to areas of small irregular defects. Increasing the magnification to 10000X revealed a linear roughness pattern in both groups before aging, which was multidirectional in Group UT and unidirectional in Group HT. However, after aging Group UT showed a change in the roughness pattern to become multidirectional with an increase in the number of grooves, in addition to a large circular defect, whereas, Group HT showed an increase in the number of grooves while maintain the same pattern, with multiple small circular defects. (Figure 3)

Color stability results:

Results showed a significant difference between the tested groups, where Group HT was higher than Group UT. (Table 3)

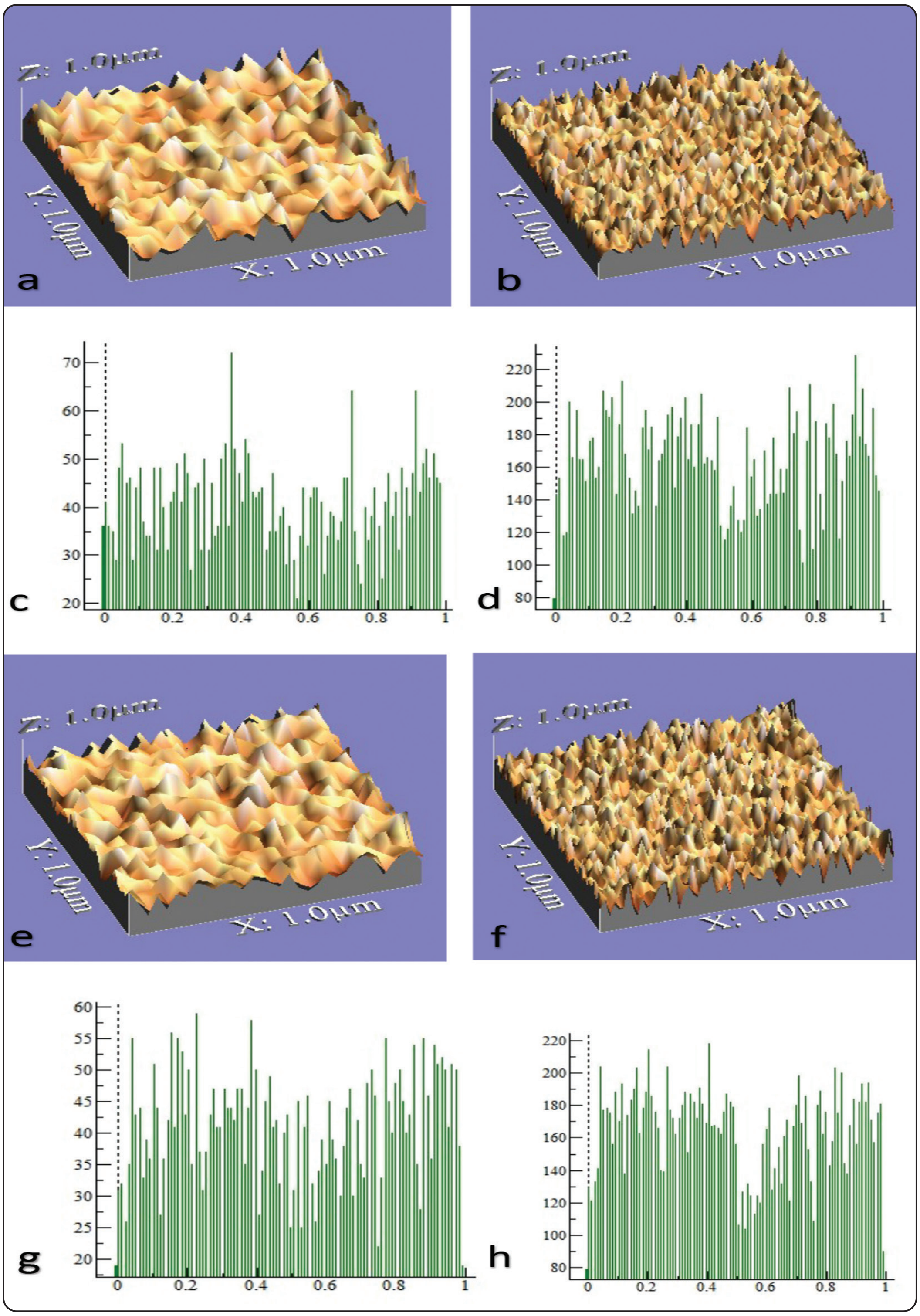

Fig. (2) 3D optical images and histograms of surface roughness (a,c: UT before aging; b,d: UT after aging) and (e,g: HT before aging; f,h: HT after aging)

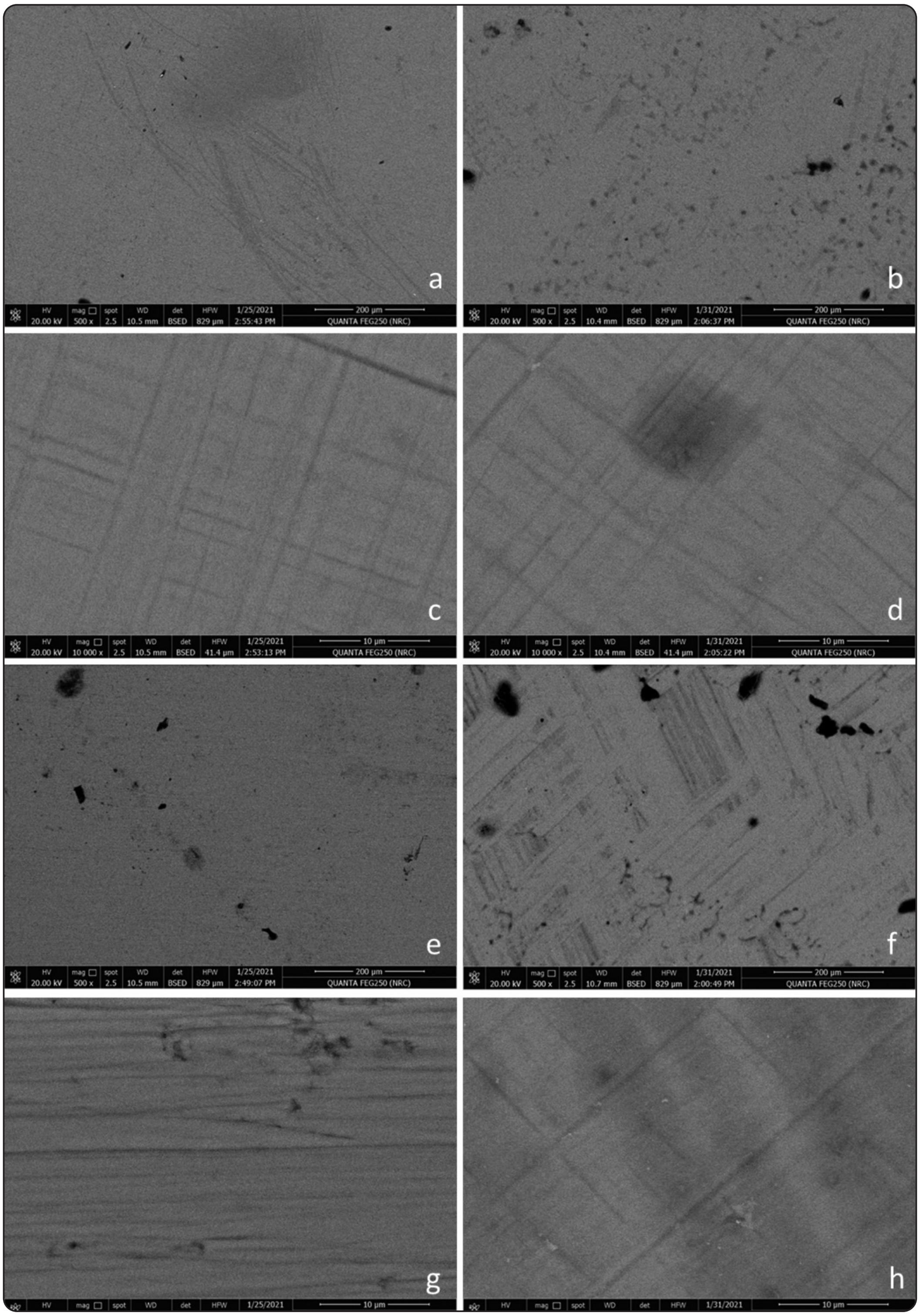

Fig. (3) SEM images of surface roughness; a: UT at 500x before aging, b: UT at 500x after aging, c: UT at 10000x before aging, d: UT at 10000x after aging, e: HT at 500x before aging, f: HT at 500x after aging, g: HT at 10000x before aging, h: HT at 10000x after aging.

DISCUSSION

The results rejected the null hypothesis, as there was a statistically significant effect of accelerated aging on surface roughness of UTML and HTML, where the surface roughness increased after aging in both groups. In addition, both groups showed changes in the color parameters after aging.

In the present study, ultra-translucent (UT) multilayered zirconia (5Y-PSZ), which contained high amount of cubic phase (71wt%) and 9.55% yttrium was tested [17]. It was claimed that UT zirconia combined the aesthetic performance of glass ceramics, while possessing higher flexural strength and fracture toughness compared to lithium disilicate [18].

High-translucent (HT) multilayered zirconia (3Y-PSZ) was also tested. It contained a considerable amount of cubic phase (41wt%), yet lower that UT zirconia. It is stabilized with 5.2% yttrium, which is also lower than that in UT zirconia. It offered high fracture toughness and flexural strength values compared to conventional zirconia [19]. Both tested materials exhibited favorable mechanical and esthetic properties at a thin conservative thickness [7].

In the present study, disc shaped specimens were constructed with 11mm diameter to include the whole thickness of the multilayered blanks with all its layers, to provide sufficient area for testing and to allowed easy handling during polishing and testing [20].

Discs thickness was 0.6mm to simulate the thickness used in laminate veneers restorations as recommended by manufacturers of both tested materials. Variable thickness was avoided to eliminate any confounding factor that might affect the outcomes tested. Shade A2 was selected to correspond to the most prevalent tooth shade among the population [21].

The sintering temperature was precisely followed to eliminate any changes in the microstructure of both materials that might affect the tested outcomes, as increasing sintering time would increase the cubic content and the grain size which would affect the color and translucency [8].

Polishing was performed rather than glazing, as it was found that polishing produces a smoother surface with less abrasion for antagonist enamel surface [5]. The glaze layer quickly wears away leaving rough surface of unpolished ceramic causing antagonist wear [4,10].

Aging was performed to simulate 15-years of intra-oral service, to attain stronger evidence of the effect of aging and to show the long-term behavior of the tested materials [22].

The Ra was used in the present investigation, as it is a reliable index of surface roughness, commonly used for roughness determination and its use allowed easier comparability to other studies[23].

Scanning electron microscope was used to complement the qualitative analysis of the surface roughness to obtain a high-quality real-time image of the specimen surface [24].

A black background was utilized in color measurement, because it showed the lowest acceptability threshold values, allowing the evaluation of slight color differences [25].

Regarding surface roughness results: A significant increase in surface roughness after aging was seen in both groups. This might be attributed to the fact that aging or low temperature degradation of translucent zirconia is associated with spontaneous transformation of the metastable tetragonal phase to monoclinic phase [26] with loss of small-sized external zirconia grains and an expansion in the existing surface grains, inducing irregularities, hence increasing the surface roughness [27].

These results came in agreement with **Toma et al. (2022)** who showed that micro-roughness of 4Y-TZP and 5Y-TZP increased after aging. Our results were also supported by **Amin & Etreby, (2020)**[28] finding, where there was a statistically significant increase in mean Ra after aging in (5Y-TZP) cubic zirconia. However, our results did not coincide with **Kou et al. (2019)** and **Chaimongkon Peampring et al., (2021**), who found no significant increase in surface roughness in 5Y-PSZ zirconia materials after artificial aging for 10-hours.

However, as shown in our results, the surface roughness after aging for UTML and HTML was 0.2533µm and 0.2536µm respectively, both below the roughness threshold detectable by the tongue (0.5µm) **Chaimongkon Peampring et al., (2021**).

With regards to the results of the effect of aging on surface roughness of UTML versus HTML zirconia discs, a significance difference was found in the tested groups before and after aging, which could be attributed to the difference in the material composition. The effect of aging was more detected in (HT) Group. This could be attributed to the fact that the material with 3 mol% Yttrium content (HTML) underwent a more intense aging process accompanied by phase transformation, volume increase of particles, grain detachment, and surface roughness [8]. For UTML (5 mol% Yttrium), the surface roughness after aging showed a minor increase, meaning that UTML zirconia was not much affected by aging with a more stable crystal structure.

These results partially agreed with the results of the study conducted by **Peampring & Kengtanyakich (2021**), who found that hydrothermal aging resulted in surface alteration of non-cubic translucent zirconia and cubic-containing zirconia which consisted of less than 30% of cubic crystals (3Y-PSZ) in comparison to (5Y-PSZ), phase transformation would not occur after being accelerated by hydrothermal aging. The difference in the results might be due to the fact that the above-mentioned study used different commercial brands with thicker samples (1mm).

Regarding color stability results: Both groups showed changes in the color parameters after aging. This might be due to the breakdown of the metal oxide content of the colorants added to the ceramic itself. The breakdown of metal oxides is followed by peroxide compound formation that would likely change the color of the shaded ceramic material [10]**.**

The largest amount of color change was observed in HT group, this could also be due to the difference in the materials composition, with HTML (3Y-PSZ) having the lowest amount of cubic phase (41wt%).

Perceptibility and acceptability are the most often used threshold units in the field of color science [29]. When the color difference (ΔE) between compared objects is seen by 50% of observers (the other 50% will notice no difference), this is 50:50% perceptibility threshold. When color difference is considered acceptable by 50% of observers (the other 50% would consider it unacceptable), this corresponds to 50:50% acceptability threshold [30]. It is reported that a ΔE of 2 is the 50:50 perceptibility threshold, whereas a ΔE of 3.7 was found to be the 50:50 acceptability thresholds [31].

The amount of color change in each group was above the level of perceptibility (ΔE=2), indicating that it was perceivable to the human eye, however it was still within the level of clinical acceptability (ΔE=3.7) as determined by many studies [10].

The increased color change might be also attributed to subjecting both surfaces of the samples to aging, which might have aggravated the effect of aging; however, this does not occur in the clinical situation[6] , where the restoration's fitting surface is protected by the cement layer. It might also be due to testing thin thickness specimens, as it is well known that decreasing the specimens thickness, increased the adverse effect of aging on color stability [10] and [19].

The increased color change could be due to increase in surface roughness, that cause restoration discoloration, staining, and plaque and calculus formation with subsequent periodontal structures compromise [(5)].

These results come in agreement with **Hamza et al. (2017)** and **Koseoglu et al. (2020),** who reported an increase in color change after continued aging of HTML zirconia. However, our results did not agree with the study of **Ahmed, et al. (2020)**, who reported that Ultra-translucent cubic zirconia had insignificant color change after aging (DE=1.41±0.63). The difference in the results might be attributed to using a thicker sample (1.5mm) and different commercial products (DD cube X^2) **putra et al., (2017)**.

It is recommended to test the microstructure to demonstrate the aging dynamics, test the effect of different thicknesses and different beverages on long-term behavior of UTML and HTML, finally conduct more studies comparing the behaviors of UTML and HTML to other ceramics with different polishing and glazing parameters.

CONCLUSIONS

Within the limitations of the present study, the following can be concluded:

1. Accelerated aging produced higher surface roughness in HTML compared to UTML.
2. Color stability of UTML and HTML was affected by accelerated aging, but the change was within the clinically accepted limit.
3. UTML has more resistance to accelerated aging compared to HTML.

Clinical implications:

When constructing laminate veneers restorations, UTML is considered more stable than HTML in terms of surface roughness and color stability.

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CONFLICT OF INTEREST

The authors have no conflicts of interest to declare.

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