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Optical Properties and Flexural Strength of Artificially Aged Tetragonal/Cubic Ultra-Translucent Zirconia

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Optical Properties and Flexural Strength of Artificially Aged Tetragonal/Cubic Ultra-Translucent Zirconia

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ABSTRACT

Purpose: The present study aimed to evaluate the effect of artificial aging on the optical properties and flexural strength of ultra-translucent tetragonal/cubic zirconia. **Materials and Methods**: A total of 30 (N=30) ultra-translucent cubic zirconia discs (DD cube X^2) were fabricated. Samples were divided into 3 equal groups (n=10) according to the aging protocol followed including Group (1): (control), samples were not subjected to hydrothermal aging protocol, (n=10), Group (2): Samples were subjected to one-hour hydrothermal aging protocol, (n=10), Group (3): Samples were subjected to 3 hours hydrothermal aging protocol, (n=10). Samples of each group were subjected to optical properties determination using spectrophotometer including (Translucency parameter TP, Contrast ratio CR, Change in color ΔE) and biaxial flexural strength determination using universal testing machine. Obtained values were statistically analyzed. **Results**: Statistical analysis using ANOVA test revealed no significant difference between the three tested groups. The highest mean value of translucency and biaxial flexural strength were recorded at the control group. While group (3) samples recorded the lowest mean values. Color change (ΔE) was performed for groups (2 and 3) to detect the amount of color change from control group. Group (3) samples recorded higher mean values than group (2) samples. **Conclusions**: Hydrothermal aging did not affect the optical properties and flexural strength of tetragonal / cubic zirconia (DD cube X^2).

KEYWORDS

Biaxial flexural strength, cubic zirconia, hydrothermal aging, translucency.

INTRODUCTION

Dental ceramics are recognized for their excellent aesthetics and biocompatibility [(1)]. Zirconia-based ceramics are categorized as one of the most common and the most used ceramics in the contemporary

- Paper extracted from Master thesis titled "Optical Properties and Flexural Strength of Artificially Aged Tetragonal/Cubic Ultra-Translucent Zirconia".

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dental practice due to their super mechanical properties and outstanding biocompatibility [2].

However, cohesive chipping of the ceramic veneer is observed as a frequently clinical complications of zirconia restorations [3]. Also, the veneering process is taking too much time and critical procedure [4].

However, with continuous evolution in computer aided design- computer aided manufacture CAD-CAM technology, it become available to design and fabricating of monolithic restorations and by virtue of the consistent advancement of new translucent Y-TZP, new CAD-CAM systems improve the manufacturing of monolithic Y-TZP crowns and FDPs with the objective to put an end to the issue of veneering porcelain's chipping [5].

Recently, cubic zirconia had been introduced to the dental field. This new generation of zirconia has excellent optical features compared to the other types of zirconia.

Increasing yttria content of zirconia to more than 8 mol% will help in stabilizing the cubic stage inside the plan. Recently, different types of "high – translucent" or cubic containing" zirconia have emerged in the market, representing around 8 mol% to 10 mol% yttria [6].

In 2015, two new products were introduced based on cubic zirconia formulation: cubic ultra-translucent (UT) and super-translucent (ST) zirconia. Both products are available in a multilayered version (UTML and STML). The flexural strength of UT zirconia is 550 MPa, while that of ST zirconia is 750 MPa [7].

However, previous studies revealed that, when the zirconia surface is in contact with humid environment, it can suffer a slow, spontaneous and progressive phase transformation [8].

Such early catastrophes were attributed to hydrothermal degradation or low-temperature degradation (LTD) phenomenon identified medically as aging of zirconia [9] and take place when zirconia is processed in a humid medium.

Furthermore, disadvantages of the LTD can be represented in advanced wear degrees with loss of little external zirconia grains in the adjacent media producing an expansion in the surface inspires irregularity and uplift with both mechanical and aesthetic deterioration [10].

The null hypotheses proposed for the present study were that artificial aging will not affect optical properties and flexural strength of cubic/tetragonal zirconia.

MATERIALS AND METHODS

Preparation of samples:

Using CAD/CAM milling machine blank of DD cube X^2 zirconia was milled into cylinders of 12.5 mm diameter. The blank was inserted into CAD/CAM Roland machine (Roland, japan). and then sectioned into 1.8mm discs using diamond micro-saw (IsoMet 4000 precision cut, Buehler USA).

The proportions of each sectioned sample were checked with a digital caliper (Mituoyo, Japan). Partially sintered disc samples were fully sintered according to the constructor's references in high-temperature furnace (TABEO -1/S/ZIRKON-100, Mihomvogot, Germany). The final measurements of the samples were: 10±0.05 mm in diameter and 1.5±0.05 mm in thickness.

Minimal finishing procedure was done agreeing with the constructor's guidelines. The finishing procedure included using diamond finishing stone at low speed using low pressure twice on each surface of the sample in one direction.

Polishing of samples was carried out using finishing and polishing kit (KENDA, Vaduz, lichenstien), mounted in a low speed hand piece without using polishing paste.

Classification of samples:

A total of 30 (N=30) ultra-translucent cubic zirconia discs were made-up. Samples were distributed into 3 equal groups (n=10) along with the aging protocol followed:

1. Group (1): (control), samples were not subjected to hydrothermal aging protocol, (n=10).
2. Group (2): Samples were subjected to one-hour hydrothermal aging protocol, (n=10).
3. Group (3): Samples were subjected to 3 hours hydrothermal aging protocol, (n=10).

Hydrothermal aging of the samples:

Samples of groups (2) and (3) were subjected to hydrothermal aging procedure using an autoclave (TS-Tau sterile autoclave, Fino Moransco-COMO, Italy). Samples were packed independently in a small sterilization pack and labeled, then arranged on autoclave trays.

The autoclave was programmed to a temperature of 134oC and under 2 bar pressure. Samples of group (2) were placed in the autoclave for one hour while samples of group (3) were placed for three hours.

After cooling to room temperature, samples of each group were stored in a glass air-tight container till testing.

Testing Procedures:

Optical properties:

Translucency determination (TP value):

Samples' translucency was measured by a movable reflective spectrophotometer. The aperture size was set to 4 mm and the samples were exactly aligned with the device. The samples were placed in the center of the measuring port and were kept in the same position for the white and black backings.

Contrast Ratio (CR) value:

Contrast ratio of all samples was measured using Reflective Spectrophotometer. The opening was set to 4 mm and the samples were strictly leveled through the device. The measurements were taken at the center of each sample over a white and black backings.

Change in color measurements (*Δ*E):

Reflective spectrophotometer was used to measure samples change in color. The aperture size was 4 mm aligning the samples with the device. A white background was appointed and measurements were prepared along with the CIE L*a*b* color space referring to the CIE standard illuminant D65.

Biaxial Flexural Strength (MPa):

Biaxial flexural strength tests were performed using Bluehill Lite Software from Instron. Testing was done with a computer-controlled materials' testing machine with a loadcell of 5kN, at a cross-head speed of 1 mm/min. Data were recorded using computer software. Discs were placed on 8-mm diameter circular knife-edge support and loaded centrally with a spherical indenter of 3.8-mm diameter. A thin sheet of tin foil was placed between each sample and load applicator tip to facilitate a uniform distribution of the load. The higher the fracture stress, the more elastic energy stored shorty before discharged on fracture making a greater number of fragments.

Statistical analysis

Data expressed in mean and standard deviation. One-way ANOVA was used for intergroup evaluations. Statistical analysis was done on Version 25 of IBM* SPSS® Statistics.

RESULTS

Statistical analysis of translucency parameter (TP) values:

Group (I) samples recorded the highest mean values (11.17±0.33), followed by group (II) samples

(10.98±0.39). While group (III) samples recorded the lowest mean values (10.88±0.41).

Statistical analysis using ANOVA test revealed no significant difference between the three tested groups (Table 1).

Table (1): *Mean values, standard deviation (SD) and ANOVA test of translucency parameter (TP) for dissimilar group*

Groups	Group (I) (control), samples were not subjected to hydrothermal aging protocol	Group (II) Samples were subjected to 1hour hydrothermal aging protocol	Group (III) Samples were subjected to 3 hours hydrothermal aging protocol
N_O of samples	10	10	10
Mean	11.17	10.98	10.88
SD	0.33	0.35	0.41
P Value	0.334*		

*SD = Standard deviation, P= probability, * non-significant at p>0.05*

Statistical analysis of contrast ratio (CR) values:

Group (I) samples recorded the highest mean values (0.708±0.007), followed by group (II) and (III) samples with equal mean values (0.709±0.008). Statistical analysis using ANOVA test revealed insignificant difference between groups (Table 2).

Statistical analysis of change of color from control group (ΔE) values:

Group (III) samples recorded higher mean values (1.41±0.63) than group (II) (1.05±0.54). Statistical analysis using ANOVA test revealed insignificant difference between groups (Table 3).

Table (3): Mean values, standard deviation (SD) and ANOVA test of change of color from control group (ΔE) for different groups

Groups	Group (II) samples were subjected to 1hour hydrothermal aging protocol	Group (III) Samples were subjected to 3hours hydrothermal aging protocol
N_O of samples	10	10
Mean	1.05	1.41
SD	0.54	0.63
P Value	0.227*	

*SD = Standard deviation, P= probability, * non-significant at p>0.05*

Statistical analysis of Biaxial flexural strength values (MPa):

Group (I) samples recorded the highest mean values (665.38±196.59), followed by group (II) samples (613.56±134.80). While group (III) samples recorded the lowest mean values (599.27±142.52). Statistical analysis using ANOVA test revealed insignificant difference between groups (Table 4).

Table (2): *Mean values, standard deviation (SD) and ANOVA test of contrast ratio for different groups*

Groups	Group (I) (control), samples were not subjected to hydrothermal aging protocol	Group (II) Samples were subjected to 1hour hydrothermal aging protocol	Group (III) Samples were subjected to 3 hours hydrothermal aging protocol
N_O of samples	10	10	10
Mean	0.708	0.709	0.709
SD	0.007	0.008	0.008
P Value	0.966*		

*SD = Standard deviation, P= probability, * non-significant at p>0.05*

Table (4): Mean values, standard deviation (SD) and AVOVA test of flexural strength (MPa) for different groups

Groups	Group (I) (control), samples were not subjected to hydrothermal aging protocol	Group (II) Samples were subjected to 1hour hydrothermal aging protocol	Group (III) Samples were subjected to 3 hours hydrothermal aging protocol
N_O of samples	10	10	10
Mean	665.38	613.56	599.27
SD	196.59	134.80	142.52
P Value	0.820*		

SD = Standard deviation, P= probability,
** non-significant at p>0.05*

DISCUSSION

Aging of zirconia or LTD is a phenomenon resulting from its contact with humid environment, leading to the transformation of crystals from tetragonal to monoclinic phase (t-m) primary taking place on the exterior of the zirconia. Aging of zirconia or low thermal degradation (LTD) negatively influences the mechanical properties of the material [11].

DD Cubex2 blank was used in this study representing cubic zirconia as the manufacturer of this zirconia system claims that this type of cubic zirconia is based on 53% cubic and 47% tetragonal structure.

Cubic zirconia blanks were cut using a low speed diamond saw to an approximate oversize in thickness of 20%-25% (thickness of 1.8mm) to compensate for the expected sintering shrinkage.

Sintering of the samples were done following the manufacturer's recommendations at high temperature furnace at 1450-1550^{0}C to produce impenetrable structures confirming unity between the zirconia grains and to avoid larger grain formation.

Finishing and polishing of constructed samples was done in order to simulate real clinical conditions as dissimilar finishing procedures have been stated to have a major impact on the mechanical properties of zirconia [12]. Both procedures were carried out for all samples by the same operator, in one direction and for the same time of application for each sample.

Autoclaving was recommended in several studies as an established method for accelerated aging of Y-TZP materials as it induces low temperature degradation (LTD) in zirconia restorations [13,14]. Samples in group (2) and (3) in the present study received different aging protocols simulating different time intervals. Aging in an autoclave for 1 hour at 134^{o}C was done for group (2) samples[15], while aging in an autoclave for an additional 2 hours at 134^{o}C, totaling 3 hours was done for group (3) samples [16].

The approach followed in the present study is in accordance to Chevalier and colleagues [17] who considered the handling of zirconia samples in an autoclave for 1 hour at 134^{o}C is equivalent to 3-4 years in-vivo aging. While for group (3) samples; the aging procedure followed simulate approximately 9–12 years of clinical use.

The use of reflective spectrophotometer for obtaining the CIELAB coordinates is common in the field of dental investigation; accordingly, it was used in the present study to obtain ΔE, CR and TP for samples of different groups.

In addition, the biaxial flexure test is used for testing strength of brittle materials. The biaxial flexure test using uniform pressure on disc with a ball on ring fixture was conducted in this study.

The results of this study revealed that there was insignificant difference in optical properties of DD Cube x2 samples of different groups. Artificial aging using the two tested procedures (groups (2) and (3)) did not have emotional impact on the optical properties of the samples. Accordingly; the null hypotheses that there would be no difference in translucency of tetragonal/cubic zirconia after hydrothermal aging was accepted.

These results are in accordance with the results obtained in other studies (18,19). In these studies, hydrothermal aging has no effect on the (TP) of tested zirconia samples.

The translucency didn't decrease after aging in the tested type of cubic zirconia which is probably associated with the decrease in the amount of transformation of zirconia from the tetragonal to the monoclinic phase, and this is accompanied by decreased superficial irregularity, light scattering, and reflection (15). DD cube X2 showed no change in TP and CR after aging for the reason that of the least change in superficial irregularity triggered by means of LTD.

In addition, LTD at all times begins at the surface of the sample and in the present study the surface of the 1.5 mm thick samples used has a less proportion of the whole volume of each sample thus consequently, would have a lesser effect than it does in thinner zirconia structures (18).

Moreover, because of the decrease in the transformation from tetragonal to monoclinic phase, microcracks and superficial porosity could also be diminished resulting in no change in translucency. furthermore, porosity is one of the main factors which has impact on the translucency of ceramics after aging as seen by some authors (20).

Change the color of zirconia observed next to frequently repeated steam autoclave aging may be caused by pigment cessation during frequent heat exposure and color variability (21). The breakdown of metal oxides during aging lead to the formation of peroxide compound which could change the color of the shaded ceramics.

In addition, the DD cube X2 zirconia had insignificant color change after aging and was below equally clinical acceptability and perceptibility levels (mean ΔE values were below 3 in both groups). The finding was in consistent with previous study (19) which reported that the reason for the resistance of DD cube X2 to change in color after aging may be due to the presence of orange coloring metallic oxide which has the greatest color stability throughout changed aging times.

On the other hand, these finding are in contradiction with the results reached by another study (22) in which accelerated aging had an effect on color and translucency of different zirconia brands. The difference in results may be attributed to the different methodology of both studies. The later study (22) was conducted with different aging time (5 hours) and different thickness of the samples (0.5mm). It was previously explained that light reflectance increases when the thickness of the samples decreased as the thickness of the material has a major influence on translucency.

Regarding biaxial flexural strength there was insignificant effect on flexural strength could be observed in the results of the present study.

This can be clarified by that transformation zone has no deeply enough extension into the material to impact its main part strength. This clarification is reinforced by that the tetragonal to monoclinic phase transformation takes place at the surface and continues into the material, with the primarily transferred fragments generating first surface uplifts, because of volume increases, without creating noticeable internal stresses (23). However, this was not the situation in the present experiment.

Our results in this respect conform to the reports of previous other studies (24,25) who reported that aging did not affect strength even after prolonged durations.

It was previously stated that highly translucent zirconia with at least 5.5 mol.% yttria do not undergo hydrothermal degradation even after 54 hours of artificial aging (26). consequently, these materials are totally unaffected by aging under in vitro circumstances without application of external load.

As the material used in this study had high Yttrium content (>5 mol% according to the manufacturer), a low susceptibility to LTD might be predictable.

One more main fact to highlight is that tetragonal to monoclinic phase transformation is dependent on the addition of alumina. This could possibly a justification for the greater confrontation to degradation by prevention of propagation of monoclinic transformation, since tetragonal zirconia is in general supersaturated with alumina to rise its aging performance and strengthen the grain margins[27].

The previously mentioned fact that in cubic zirconia, cubic grains coexist with tetragonal grains can explain the biaxial flexural strength results obtained in the present study where the cubic grains are less vulnerable to low thermal degradation and this is due to: higher concentration of oxygen vacancies in grain boundaries than that of tetragonal zirconia, the grain boundaries of cubic zirconia which are less vulnerable to water attack than those of tetragonal zirconia, much larger grain size of cubic zirconia than tetragonal zirconia and high yttria stabilizer content which is the main cause of suppressing the degradation.

On the other hand, results obtained disagree with those of a previous study which reported that aging decrease flexural strength of cubic zirconia [28]. This difference may be due to different design and methods of the studies.

The present study is not free from limitations. Among these limitations is that only one cubic zirconia brand was investigated. Other high translucent zirconia with different compositions available in the market may act differently under simulated oral ageing.

Another limitation in this study is relying on disk-shaped samples to evaluate the material properties rather than more clinically relevant restorations and different prosthetic designs.

CONCLUSIONS

The following can be concluded from the present study:

1. Hydrothermal aging has no effect on the optical properties of tetragonal / cubic zirconia (DD cube X2).
2. Hydrothermal aging has no effect on the flexural strength of tetragonal / cubic zirconia (DD cube X2).

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