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Effects of Hydrothermal Treatment on the Phase Transformation, Surface Roughness, and Mechanical Properties of Monolithic Translucent Zirconia

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Clinical Relevance

Although the mechanical properties of translucent monolithic Y-TZP ceramics were found to be related to the coloring and material types, minimal effects of hydrothermal treatment on their phase transformation, surface roughness, and mechanical properties were observed.

SUMMARY

Objectives: This study aimed to investigate the effects of hydrothermal treatment on four types of monolithic, translucent, yttria-stabilized, tetragonal zirconia polycrystals (Y-TZPs).

Methods and Materials: Two commercially available Y-TZP brands—SuperfectZir High Translucency (Aidite Technology Co, China) and Katana HT (Kuraray Noritake Dental, Japan) were assessed. For each brand of Y-TZP, materials

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of four coloring types, including noncolored (NC), colored by staining (CS), precolored (PC), and multilayered (ML) specimens were investigated after hydrothermal aging in an autoclave at 134°C/0.2 MPa for 0 (control group), 5, 10, and 20 hours. The tetragonal-to-monoclinic phase transformation, surface roughness, flexural strength, and structural reliability (Weibull analysis) were measured and statistically analyzed (α=0.05). The subsurface microstructure was analyzed with scanning electron microscopy.

Results: The group ML exhibited the lowest flexural strength and Weibull characteristic strength among the four coloring types (p<0.05). Slight increases in the monoclinic phase volume, flexural strength, and Weibull characteristic strength were observed after hydrothermal aging (p_{all}<0.05). Regardless of coloring type, no significant effects of aging on the Weibull modulus or surface roughness were found for the tested materials. Compared with the Katana HT cross-sections, the SuperfectZir High Translucency cross-sections exhibited a similar but thicker transformation zone.

Conclusions: The coloring procedure and material type were found to affect the mechanical properties and aging resistance of translucent monolithic Y-TZP ceramics. Regardless of the aging time, the surface roughness of the tested Y-TZP ceramics remained unchanged.

INTRODUCTION

With the development of computer-aided design–computer-aided manufacturing (CAD–CAM), monolithic restorations are increasingly being promoted to avoid the cohesive fracture of veneering porcelain (chipping) and facilitate a more conservative tooth preparation.[1,2] Zirconia has been a dominant ceramic, typically fabricated in monolithic form, for a wide range of clinical applications due to its excellent mechanical properties.[3-5] At ambient pressure, zirconia has three crystalline phases: tetragonal (*t*), monoclinic (*m*), and cubic (*c*).[6,7] For biomedical applications, *t*-phase zirconia is typically stabilized by adding yttria (Y_2O_3) to pure zirconia to achieve enhanced mechanical properties, which is known as yttria-stabilized tetragonal zirconia polycrystal (Y-TZP).[8,9] Among all the available dental Y-TZP ceramics, three mol% (5.2 wt%) yttria is the most common dopant for stabilizing zirconia and is denoted as 3Y-TZP. However, the major drawback of the first generation of 3Y-TZP is its high opacity, especially when compared with glass ceramics.[7] To reduce the opacity of Y-TZP, various approaches have been proposed, including the reduction of an alumina additive, the elimination of porosity, an increase in the sintering density, and an increase in the *c*-phase content.[7,9-11] The *c*-phase of zirconia is isotropic in different crystallographic directions, which decreases light scattering at grain boundaries. The *c*-phase attracts yttria and leaves the *t*-phase prone to spontaneous transformation. Therefore, researchers have attempted to stabilize pure zirconia with a significant amount of *c*-phase by increasing the yttria content.[12] The latest translucent Y-TZP contains a higher concentration of yttria (4Y-TZP or 5Y-TZP) than that of 3Y-TZP, and they exhibit comparable translucency with lithium disilicate.[7,9,10] These materials are typically used for anterior crowns and fixed dental prostheses (FDPs). Improved translucency of 5Y-TZP and 4Y-TZP has been achieved by increasing the amount of transparent-phase (*c*-phase) zirconia; however, the mechanical strength decreases in this case.[10,13] A dental laboratory survey of 39,287 restoration records reported a failure rate of 2.06% over 5 years for anterior monolithic restorations.[14] The second generation of 3Y-TZPs (translucent 3Y-TZPs), which have been refined by reducing the amount of alumina additive and decreasing the grain size and sintering porosity, has been considered to strike a suitable balance between esthetics (color and translucency) and function (strength).[7,9,15] From a clinical point of view, translucent 3Y-TZPs are the most widely used, and they are considered the proper dental ceramic for monolithic restorations.[7,16,17]

Conventionally, Y-TZPs are produced with uniform translucency and color (single-layered).[18] To change the basic color of Y-TZP (white to ivory) to a natural tooth color, two main approaches for coloring Y-TZP are available: precoloring and coloring by staining.[19-21] For precolored Y-TZP, metal oxides capable of reproducing a color are added into the starting powder. The colored-by-staining technique involves dipping the noncolored Y-TZP in coloring liquids or painting the coloring liquids on with a brush. These two coloring techniques have been proven to successfully reproduce the color of human teeth, although conflicting results have been reported regarding the effects of coloring procedures on the flexural strength of Y-TZPs. Ban and others[18] observed a reduction in the flexural strength and fracture toughness of Y-TZPs colored by staining in a coloring liquid containing erbium (Er) and neodymium (Nd) ions. In contrast, other studies reported that the coloring procedure exhibited no effects on the flexural strength of Y-TZPs.[6,22,23] The lack of consensus may be due to the different Y-TZPs and coloring liquids adopted in previous studies. Recently, multilayered Y-TZPs with gradual changes in color and translucency have been introduced commercially. Multilayered Y-TZPs normally include three to four layers (enamel, dentin, and transition layers) corresponding to the transition from enamel to dentin.[9,22] Although manufacturers claim that multilayered Y-TZP blocks have a flexural strength similar to that of conventional single-layered products,[24] some authors have reported results that conflict with the internal data of these manufacturers.[13,15] Consequently, thorough investigations are additionally needed to provide clinical recommendations.

Theoretically, all types of 3Y-TZPs undergo a *t*- to *m*-phase transformation when exposed to stress in the oral environment.[7,25,26] The phase transformation leads to a volumetric expansion that can stop crack propagation and results in the superior mechanical properties of 3Y-TZPs (transformation toughening). However, a slow *t*- to *m*-phase transformation can also occur without stress under moist conditions and at body temperature, which is referred to as low-temperature degradation (LTD) or aging.[27,28] Although limited evidence of LTD is available in clinical situations, LTD leads to a slow phase transformation, which reduces the flexural strength of 3Y-TZPs under laboratory settings.[3,16] The effects of different coloring procedures (precoloring vs coloring by staining) on the aging behavior of 3Y-TZPs have been investigated in a previous study.[19] After hydrothermal aging, the precolored 3Y-TZP exhibited significantly greater phase transformation than the colored-by-staining 3Y-TZP. Given that a small deviation in composition, structure, and fabrication can make a huge difference,[9,25,29] it is critical to evaluate and compare the mechanical and physical properties of different types of 3Y-TZPs after aging in a comprehensive manner. However, to the best of the authors' knowledge, limited information is available.

Therefore, this study aimed to evaluate the surface roughness, subsurface microstructure, flexural strength, structural reliability (Weibull analysis), and *t*- to *m*-phase transformation of four coloring types of second generation 3Y-TZPs (noncolored, precolored, colored by staining, and multilayered) produced by two commercially available brands (SuperfectZir High Translucency and Katana HT). The null study hypotheses were as follows: 1) that the four coloring types of 3Y-TZPs would exhibit similar mechanical and physical properties, and 2) that the four coloring types of 3Y-TZPs would behave similarly after hydrothermal aging.

METHODS AND MATERIALS

Specimen Preparation

Two commercial brands of translucent 3Y-TZPs (SuperfectZir High Translucency, Aidite Technology Co, Qinhuangdao, China; Katana HT, Kuraray Noritake Dental, Niigata, Japan) were investigated in the present study. For each brand, four coloring types of products were employed: noncolored (NC), colored by staining (CS), precolored (PC), and multilayered (ML). The characteristics of the investigated 3Y-TZPs are listed in Table 1.

Table 1: *Characteristics of the Materials Used in this Study*

Material	Type of Product	Chemical Components (wt%)	Manufacturer	Sintering Conditions		
				Heating	Dwelling Time	Cooling
SuperfectZir High Translucency	NC	ZrO_2+HfO_2:92%-96%; Y_2O_3: 5.3%; Al_2O_3: 0.25 wt%	Aidite Technology Co., Qinhuangdao, China	7°C/min to 900°C + 5°C/min to 1530°C	2 h	10°C/min to room temperature
SuperfectZir High Translucency A2	PC					
SuperfectZir High TranslucencyMultilayer	ML					
Katana HT10	NC	ZrO_2+HfO_2:90%-95%; Y_2O_3: 5%-6%; other oxide: 0-2 wt%	Kuraray Noritake Dental, Niigata, Japan	10°C/min to 1500°C	2 h	10°C/min to room temperature
Katana HT12	PC					
Katana HTML	ML					

Abbreviations: ML, multilayered; NC, noncolored; PC, precolored.

Bar-shaped specimens were fabricated using a CAD–CAM milling machine (Zenotec, Wieland, Germany) from green-stage discs with compensation for shrinkage during dense sintering.[19] ML specimens were fabricated using ML discs with enamel and transition layers. During fabrication, half of the ML specimen (1.5 mm in thickness) was within the enamel layer, while the other half was within the transition layer. All four long edges of each specimen were chamfered according to ISO 14704:2016.[30] The four surfaces of the specimens were wet-polished with 600-grit and 1200-grit silicon carbide discs (Buehler, Lake Bluff, IL, USA) and ultrasonically cleaned for 15 minutes. CS specimens were colored by dipping NC specimens into a coloring liquid (zirconia coloring liquid A2, Aidite Technology Co) for 2 minutes at room temperature. For the PC and ML specimens, an A2 shade was selected. All specimens were sintered according to the respective instructions of the manufacturers. After sintering, the four surfaces of the specimens were wet polished using a polishing device (PG-1S, Biaoyu Co, Shanghai, China) with 1000-grit and 2000-grit silicon carbide discs to control the final dimensions with tolerances of ±0.01 mm (length 22 mm, width 4 mm, and thickness 3 mm).

Aging Treatment

An LTD process was simulated using hydrothermal aging, according to ISO 13356:2016.[31] The specimens of each type of 3Y-TZP were randomly divided into four groups based on aging duration (n=37). The aging treatment was performed using an autoclave (HE-50, Hirayama, Japan) at 134°C in a water vapor atmosphere at 0.2 MPa for 0 (control), 5, 10, and 20 hours.

Phase Transformation Analysis

The crystalline phases of the specimens from each group (n=5) were analyzed using an X-ray diffractometer (Empyrean, PANalytical, the Netherlands). The X-ray diffraction (XRD) patterns were recorded at 40 kV (generator voltage) and 40 mA (tube current) with Cu Kα radiation. The 2θ scan range was between 26° and 36°, with a step size of 0.01° and a scan time of 18.9 seconds/step. The monoclinic phase fraction (X_m) was calculated according to the equation proposed by Garvie and Nicholson[32]:

$$X_m = \frac{I_m(-111) + I_m(111)}{I_m(111) + I_m(-111) + I_t(101)} \quad (1)$$

where $I_m(-111)$ and $I_m(111)$ represent the monoclinic peak intensities at 2θ=28.2° and 31.4°, respectively, and $I_t(101)$ represents the tetragonal peak intensity at 2θ=29.9°.

The monoclinic volume fraction (V_m) was then calculated using a method described by Toraya and others[33]:

$$V_m = \frac{1.311X_m}{1 + 0.311X_m} \quad (2)$$

Surface Roughness Measurement

The surface roughness of the specimens was measured using a stylus profilometer (SEF 680, Kosaka Laboratory, Japan) after the respective aging treatment (n=30). The profilometer was used with a diamond stylus that moved along a length of 2.5 mm at a speed of 0.5 mm/s with a cut-off value of 0.8 mm. Four readings were taken on a single selected surface of each specimen, and the average R_a values were calculated for statistical analysis.

Flexural Strength Measurement

After the surface roughness measurement, a four-point flexure test was performed using a universal testing machine (Instron 1186, Instron, London, United Kingdom) at a crosshead speed of 1 mm/minute (n=30). The flexural strength (σ) was calculated using the following equation[34]:

$$\sigma = \frac{3Pl}{4Wb^2} \quad (3)$$

where P is the loading load, l is the length of the test span, and w and b are the width and thickness of the beam specimen, respectively.

Scanning Electron Microcopy (SEM) Observation

The cross-sectional topography patterns were examined using a scanning electron microscope (Quanta 250, FEI, USA). For each coloring type of Y-TZP ceramic, two specimens were cross-sectioned. The sections were mounted on aluminum stubs and sputter-coated with gold before being examined at an acceleration voltage of 15 kV. The images were taken at a magnification of 5000×. The depth of t- to m-phase transformation zone was estimated as described in a previous study.[35]

Energy Dispersive X-ray Spectroscopy (EDS) Measurement

EDS analysis was performed to measure the yttria contents (expressed as weight percentages) of the Y-TZPs using the same SEM instrument equipped with an energy-dispersive X-ray spectrometer. For each coloring type of Y-TZP ceramic, two green-stage specimens were fabricated for EDS measurement. For the group ML, EDS measurements were performed in each layer (enamel and transition).

Statistical Analysis

The statistical analysis was performed with the SPSS statistical software package (SPSS 19.0 for Windows, SPPS, Chicago, IL, USA). The Shapiro–Wilk test confirmed the normal distribution of the data. Three-way analysis of variance (ANOVA) with Tukey post hoc test was used to analyze the results of surface roughness, flexural strength, and V_m (α=0.05).

To determine the structural reliability, the flexural strength data were also submitted to a Weibull distribution. According to the Weibull distribution, the probability of failure (P_f) of a brittle material can be calculated with the following equation[36]:

$$P_f = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] \quad (4)$$

where m is the Weibull modulus related to the dispersion of the failure data, σ_0 is the characteristic strength representing the stress level in which 63.21% of the specimens will fail, and σ is the strength at a given P_f. The probability of failure can be estimated from the following equation[36]:

$$PP_f = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right]_f = 1 - \frac{i}{n+1} \quad (5)$$

where i is the ranking of the strength data when arranged in ascending order, and n is the number of specimens.

Using equations 4 and 5, the Weibull modulus (m) and the characteristic strength (σ_0) of the tested materials were calculated according to a method previously described in detail.[37]

RESULTS

The yttria contents for each group ranged from 5.07 to 5.75 wt% (Table 2). The R_a values of all groups are listed in Table 3. No significant differences were found between brands, among material types, or among aging durations (p=0.215, p=0.806, and p=0.106, respectively).

The V_m values of all groups are listed in Table 4. The V_m values of all groups significantly increased with aging duration (p<0.001). There were significant differences in the V_m among the material types (ML, NC, PC>CS, p<0.001) and between brands (SuperfectZir High Translucency>Katana HT, p=0.027).

The flexural strength values of all groups are listed in Table 5. The flexural strength significantly increased with aging duration (p<0.001). There were significant differences in flexural strength among the material types (CS>PC>NC>ML, p<0.001) and between brands (Katana HT>SuperfectZir High Translucency, p<0.001). For nonaged specimens, the group CS exhibited the highest flexural strength, whereas the group ML showed the lowest flexural strength.

The Weibull modulus (m) values of all groups are listed in Table 6. The Weibull modulus ranged from 9.17 to 14.98. The Weibull modulus of all groups remained unchanged after aging. No significant differences were found in the Weibull modulus between brands. In most cases, the Weibull modulus was similar among all groups.

The characteristic strength (σ_0) values of all the groups are shown in Table 7. The characteristic strength remained unchanged after aging, except for the groups CS and NC produced with SuperfectZir High Translucency. The group ML exhibited the lowest characteristic strength among the four groups.

Similar grain sizes were observed for the control specimens of different groups. Representative cross-section images of the control specimens and specimens aged for 20 hours are shown in Figure 1. The cross-sections of aged samples revealed the existence of two distinct regions: a zone near the surface that appeared relatively rough, displaying grains with sharp edges (transformed region) and a zone similar to the unaged specimens (untransformed region). After aging for 20 hours, the depth of transformation was found to be approximately 15 μm for the SuperfectZir High Translucency specimens and 6 μm for the Katana HT specimens.

Table 2: *Means and Standard Deviations (SD) of Yttria (wt%) for Each Group*

	ML	PC	CS	NC
SuperfectZir High Translucency	Enamel layer: 5.32 (0.15) Transition layer: 5.11 (0.04)	5.14 (0.18)	5.07 (0.09)	5.12 (0.13)
Katana HT	Enamel layer: 5.58 (0.18) Transition layer: 5.44 (0.25)	5.75 (0.22)	5.64 (0.16)	5.41 (0.20)

Abbreviations: ML, multilayered; PC, precolored; CS, colored by staining; NC, noncolored.

Table 3: *Means and Standard Deviations (SD) of Ra (μm) for Each Group*[a]

Aging	ML	PC	CS	NC
SuperfectZir High Translucency				
0 hour	0.29 (0.01) Aa	0.31 (0.03) Aa	0.32 (0.01) Aa	0.31(0.02) Aa
5 hours	0.31 (0.02) Aa	0.31 (0.01) Aa	0.33 (0.05) Aa	0.31(0.05) Aa
10 hours	0.32 (0.05) Aa	0.32 (0.03) Aa	0.35 (0.04) Aa	0.31(0.02) Aa
20 hours	0.31 (0.06) Aa	0.33 (0.02) Aa	0.36 (0.05) Aa	0.33(0.07) Aa
Katana HT				
0 hour	0.28 (0.02) Ab	0.30 (0.03) Ab	0.28 (0.04) Ab	0.30(0.04) Ab
5 hours	0.31 (0.04) Ab	0.39 (0.02) Ab	0.30 (0.01) Ab	0.30(0.01) Ab
10 hours	0.28 (0.01) Ab	0.31 (0.05) Ab	0.32 (0.08) Ab	0.30(0.06) Ab
20 hours	0.34 (0.03) Ab	0.32 (0.06) Ab	0.32 (0.03) Ab	0.36(0.02) Ab

Abbreviations: ML, multilayered; PC, precolored; CS, colored by staining; NC, noncolored.
[a] *Different uppercase letters in a row indicate significant differences at each time point (p<0.05). Different lowercase letters in a column indicate significant differences for different groups of each tested material (p<0.05).*

Table 4: *Means and Standard Deviations (SD) of Vm (%) for Each Group*[a]

Aging	ML	PC	CS	NC
SuperfectZir High Translucency				
0 hour	1.37 (0.79) Aa	1.40 (0.46) Aa	1.55 (0.72) Aa	0.83 (0.29) Aa
5 hours	15.94 (0.20) Ab	15.33 (0.26) Ab	14.49 (0.65) Ab	16.28 (0.26) Ab
10 hours	30.04 (0.14) Ac	29.49 (0.17) Ac	26.99 (1.00) Bc	30.46 (0.15) Ac
20 hours	38.76 (2.51) Ad	38.89 (0.15) Ad	35.50 (0.55) Ad	38.17 (2.14) Ad
Katana HT				
0 hour	1.36 (0.25) Ae	2.19 (0.61) Ae	2.13 (0.58) Ae	2.16 (0.82) Ae
5 hours	8.71 (1.03) Af	6.31 (3.69) Af	6.48 (0.76) Af	7.29 (1.05) Af
10 hours	17.99 (0.26) Ag	18.06 (0.31) Ag	14.90 (0.18) Bg	17.59 (0.75) Ag
20 hours	26.65 (0.14) Ah	26.21 (0.20) Ah	25.48 (2.54) Ah	26.36 (0.40) Ah

Abbreviations: ML, multilayered; PC, precolored; CS, colored by staining; NC, noncolored.
[a] *Different uppercase letters in a row indicate significant differences at each time point (p<0.05). Different lowercase letters in a column indicate significant differences for different groups of each tested material (p<0.05).*

DISCUSSION

A trend toward monolithic restorations has been witnessed over recent decades. Among the available materials, zirconia, specifically Y-TZP, has gained increasing popularity, and various types have been developed for monolithic use, including single- and multiple-unit restorations and implant abutments.[7,17] As one of the top material choices, 3Y-TZP ceramics have been shown to be susceptible to LTD.[16,38] The aging process may cause grain detachment and ultimately lead to strength degradation.[7,9] However, the effects of aging on 3Y-TZPs are material dependent, and studies comparing the aging behavior of different material types show conflicting results. The present study was therefore conducted to provide knowledge for developing evidence-based material selection criteria in the context of clinical practice. Based on the present findings, the null hypotheses that the four coloring types of 3Y-TZPs would exhibit similar mechanical and physical properties and that the four coloring types of 3Y-TZPs would behave similarly after hydrothermal aging were rejected.

Table 5: *Means and Standard Deviations (SD) of Flexural Strength (MPa) for Each Group*[a]

Aging	**ML**	**PC**	**CS**	**NC**
SuperfectZir High Translucency				
0 hour	656.3 (67.1) Aa	737.3 (61.1) Ba	793.8 (59.3) Ba	756.3 (65.0) Ba
5 hours	689.0 (75.6) Aa	777.9 (78.1) Ba	842.5 (86.1) Cab	797.9 (74.3) Ba
10 hours	649.6 (61.3) Aa	751.6 (70.2) Ba	881.4 (86.8) Cab	852.0 (80.2) Cb
20 hours	695.4 (69.1) Aa	769.1 (65.0) Ba	884.6 (92.8) Cb	863.1 (74.6) Cb
Katana HT				
0 hours	849.2 (72.6) Ac	961.1 (80.2) Bc	987.6 (70.9) Bc	973.0 (79.0) Bc
5 hours	882.7 (80.9) Ac	1001.8 (71.8) Bc	996.8 (76.5) Bcd	977.0 (71.3) Bc
10 hours	898.8 (73.3) Ac	1019.9 (75.4) Bc	1034.6 (65.4) Bd	981.6 (80.5) Bc
20 hours	887.9 (88.8) Ac	1018.2 (91.9) Bc	1009.6 (73.1) Bcd	963.3 (66.8) Bc

Abbreviations: ML, multilayered; PC, precolored; CS, colored by staining; NC, noncolored.
[a] *Different uppercase letters in a row indicate significant differences at each time point (p<0.05). Different lowercase letters in a column indicate significant differences for different groups of each tested material (p<0.05).*

Table 6: *Means and 95% Confidence Intervals (95% CI) of the Weibull Modulus (m) Values for All Groups*[a]

Aging	**ML**	**PC**	**CS**	**NC**
SuperfectZir High Translucency				
0 hours	10.3 (8.9- 11.7) Aa	11.2 (8.1- 14.3) Aa	13.8 (11.8- 15.9) Aa	11.8 (10.0- 13.7) Aa
5 hours	9.4 (7.6- 11.2) Aa	9.7 (7.5- 11.8) Aa	10.1 (8.7- 11.4) Aa	11.4 (9.4- 13.4) Aa
10 hours	9.2 (6.0- 12.3) Aa	10.9 (9.4- 12.5) Aa	11.0 (10.0- 12.1) Aa	11.0 (9.7- 12.4) Aa
20 hours	9.3 (6.7- 11.9) ABa	12.5 (11.2- 13.7) Ba	10.2 (9.3- 11.1) Aa	12.1 (10.9- 13.4) ABa
Katana HT				
0 hour	11.6 (9.3- 13.9) Ab	12.0 (8.7- 15.4) Ab	14.8 (13.4- 16.2) Ab	12.2 (9.7- 14.7) Ab
5 hours	11.4 (10.2- 12.6) Ab	15.0 (12.9- 17.0) Bb	13.8 (12.2- 15.3) ABb	14.5 (13.2- 15.9) Bb
10 hours	12.8 (11.2- 14.4) Ab	13.9 (11.9- 15.8) ABb	16.9 (15.2- 18.7) Bb	13.1 (11.2- 14.9) Ab
20 hours	10.5 (9.0- 12.1) Ab	11.5 (9.7- 13.2) Ab	14.0 (11.6- 16.5) Ab	14.1 (10.8- 17.4) Ab

Abbreviations: ML- multilayered; PC- precolored; CS- colored by staining- NC- noncolored.
[a] *Different uppercase letters in a row indicate significant differences at each time point (p<0.05). Different lowercase letters in a column indicate significant differences for different groups of each tested material (p<0.05).*

The four coloring types tested in this study (PC, NC, CS, and ML) cover all the available coloring options for monolithic Y-TZP ceramics. The aging treatment was performed according to ISO 13356:2015 and previous studies.[9,25,31] Based on the estimation proposed by Cattani-Lorente and others,[26] 20 hours of accelerated aging at 134°C and 0.2 MPa is estimated to correspond to 8 years *in vivo*.

Flexural strength is an important indicator for estimating the clinical performance of dental restorations.[37,39] In the present study, the flexural strength of all the tested 3Y-TZPs ranged from 656.3 to 987.6 MPa, indicating that the tested materials can be used in monolithic forms for up to three-unit FDPs.[34] However, it is unlikely to use monolithic translucent 3Y-TZPs for the application of long-span FDPs. The ML specimens exhibited the lowest flexural strength and Weibull characteristic strength among the four tested material types. The above result was in agreement with previous studies in which a significantly lower flexural strength was found for multilayered Y-TZPs than single-layered Y-TZPs.[15,22] No significant differences were observed in

Table 7: *Means and 95% Confidence Intervals (95% CI) of the Characteristic Strength (σ_0) Values for All Groups*[a]

Aging	ML	PC	CS	NC
SuperfectZir High Translucency				
0 hour	621.3 (608.4- 634.2) Aa	700.3 (671.9- 728.7) Ba	762.1 (722.7- 791.6) Ba	721.4 (704.2- 738.6) Ba
5 hours	648.7 (632.6- 664.8) Aa	735.4 (715.5- 755.3) Ba	796.5 (783.6- 809.4) Ca	759.9 (741.0- 778.7) Bb
10 hours	611.9 (583.5- 640.3) Aa	714.0 (700.0- 728.1) Ba	837.9 (828.3- 847.5) Cb	810.0 (797.4- 822.6) Dc
20 hours	655.9 (632.4- 679.4) Aa	735.9 (724.3- 747.5) Ba	836.9 (828.3- 847.5) Cb	824.6 (813.1- 836.2) Cc
Katana HT				
0 hour	809.7 (788.3- 831.1) Ad	918.3 (946.2- 985.4) Bd	951.4 (938.3- 964.5) Bd	930.2 (906.2- 954.1) Bd
5 hours	840.8 (829.6- 852.1) Ad	965.8 (961.4- 999.3) Bd	958.2 (943.7- 972.7) Bd	941.0 (928.2- 953.8) Bd
10 hours	860.8 (845.7- 876.0) Ae	980.4 (961.4- 999.4) Bd	1001.7 (984.8- 1018.7) Be	941.1 (923.6- 958.7) Cd
20 hours	841.5 (826.6- 856.5) Ade	970.3 (953.8- 989.9) Bd	970.9 (947.3- 994.4) Bde	926.5 (895.5. 957.6) Bd

Abbreviations: ML, multilayered; PC, precolored; CS, colored by staining; NC, noncolored.

[a]*Different uppercase letters in a row indicate significant differences at each time point* ($p<0.05$)*. Different lowercase letters in a column indicate significant differences for different groups of each tested material* ($p<0.05$)*.*

the flexural strength among the single-layered 3Y-TZPs that were colored using different methods. The present findings and previous reports[6,21,22] suggest that the coloring liquid does not affect the flexural strength of translucent monolithic 3Y-TZPs.

The XRD analysis showed different percentages of phase transformation among the tested 3Y-TZPs that were subjected to aging. The *t*- to *m*-phase transformation increased with hydrothermal aging. The highest V_m was found in the specimens aged for 20 hours (25.5%-38.9%), which was within the same range as the previous studies.[19,40] Among the four material types, the lowest V_m value was found in the group CS for both brands tested, which was consistent with a previous study.[19] Various factors, including particle size,[41-43] sintering protocol,[44,45] yttria content,[7,9] and coloring procedure[18,19] have been attributed to the aging resistance of monolithic Y-TZPs. The relatively

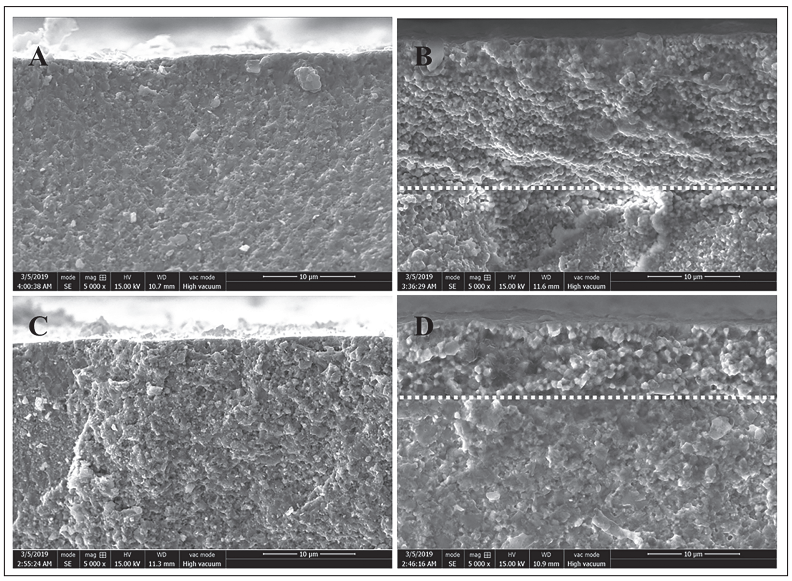

Figure 1. *Representative cross-sectional scanning electron microscope (SEM) images of the specimens at a magnification of 5000×: A. precolored specimen of SuperfectZir High Translucency aged for 0 hour (control); B. precolored specimen of SuperfectZir High Translucency aged for 20 hours; C. precolored specimen of Katana HT aged for 0 hour (control); and D. precolored specimen of Katana HT aged for 20 hours. The transformation zones are indicated by the dashed lines (B and D).*

better resistance to hydrothermal aging of the CS group may be explained by the ceria in the coloring liquid acting as a sintering aid.[19,46] Significantly, different aging resistances were also noticed between the two commercial brands, possibly due to the slightly higher yttria content in Katana HT and the different metal oxides incorporated to achieve the desired shade.[11,47] Since information about the coloring liquids and metal oxides incorporated is not available, further studies are needed to clarify these hypotheses. According to the SEM observations, the depth of transformation of the Katana HT specimens was thinner than that of the SuperfectZir High Translucency specimens (6 μm vs 15 μm), which correlated well with the V_m data and with previous studies.[19,25]

Slight but significant increases in flexural strength and characteristic strength were observed after hydrothermal aging, which was in agreement with previous studies.[40,48-50] The increase in the strength of zirconia was dependent on the percentage of the *t*- to *m*-phase transformation. The above finding indicates that the increased strength may be due to the transformation toughening mechanism, as the phase transformation on the surface of zirconia creates compressive stresses around the surface defects and further inhibits crack propagation.[51,52] However, the flexural strength values of 3Y-TZPs have also been reported to decrease[47] or remain unchanged[19,53,54] in previous studies. The conflicting findings in the literature may be due to different Y-TZP compositions, aging protocols, and flexural strength measurements. Given that only a 5% to 10% increase was found in the flexural strength of aged specimens, the present finding may not have any clinical significance. Importantly, the Weibull modulus remained unchanged after 20 hours of hydrothermal aging, indicating that the structural reliability of the tested 3Y-TZPs was stable.

Surface roughness measurements were performed as suggested by ISO 14704:2016.[30] Similar to previous reports,[16,50] aging had no effects on the surface roughness of translucent monolithic 3Y-TZPs.

The results of this study showed that the translucent monolithic 3Y-TZPs were mechanically stable and demonstrated resistance to aging; however, the mechanical properties and aging resistance were material dependent. The present study investigated specimens made from the enamel and transitional layers of multilayered zirconia. The mechanical properties and aging behavior may vary among different layers [11]. Moreover, the present findings need to be interpreted with caution, because the simulated hydrothermal aging does not include any mechanical loading that is present *in vivo*. Nevertheless, further long-term clinical studies are important for drawing solid scientific recommendations.

CONCLUSIONS

Within the limitations of the present study, the following conclusions can be drawn:

1. The mechanical properties of multilayered 3Y-TZP ceramics were significantly lower than those of 3Y-TZP ceramics of other coloring types.
2. The flexural strength and V_m of 3Y-TZP ceramics significantly increased after aging, while the surface roughness remained unchanged.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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