

**RESEARCH AND EDUCATION**

## Effect of hydrothermal aging on the optical properties of precolored dental monolithic zirconia ceramics



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Monolithic zirconia ceramics can provide excellent mechanical properties without the risk of chipping of veneering porcelain. However, low translucency and susceptibility to hydrothermal aging of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) have been reported.<sup>1</sup> When the monolithic design of Y-TZP is directly exposed to the oral environment, the absorption of water radicals can yield tensile stresses between tetragonal and monoclinic phases, and the energy difference between the 2 phases further activates tetragonal to martensitic (t→m) transformation.<sup>2,3</sup> Therefore, microstructural changes through superficial alterations resulting in surface microcracks and roughening can occur.<sup>4-8</sup> The resistance of Y-TZP to low-temperature degradation (LTD) can depend on the dwell time,<sup>9-11</sup> temperature,<sup>10</sup> grain size,<sup>9</sup> residual stress induced by surface treatments,<sup>5,6,12,13</sup> sintering processes,<sup>14</sup> stabilizing content,<sup>9,11</sup> and addition of silica.<sup>11</sup>

The effects of hydrothermal aging on the mechanical properties of Y-TZP have been investigated.<sup>15-18</sup> The flexural strength of zirconia ceramics significantly decreased after 200 hours at 134°C under 0.2 MPa.<sup>15</sup> The biaxial strength and toughness of Y-TZP decreased

### ABSTRACT

**Statement of problem.** The long-term color stability of precolored monolithic zirconia has not been thoroughly investigated.

**Purpose.** The purpose of this in vitro study was to evaluate the effect of hydrothermal aging on the optical properties, phase transformation, and surface topography of precolored monolithic zirconia ceramics.

**Material and methods.** Precolored monolithic zirconia specimens (17.0×17.0×1.5 mm, n=50) and lithium disilicate glass-ceramic specimens (16.0×16.0×1.5 mm, n=50) were artificially aged in an autoclave at 134°C under 0.2 MPa for 0, 1, 3, 5, or 10 hours (n=10). CIE Lab color parameters were obtained from spectral measurements. The translucency parameter (TP) and CIEDE2000 color differences ( $\Delta E_{00}$ ) were calculated. The microstructural and surface properties were analyzed by X-ray diffraction (XRD), atomic force microscope (AFM), and scanning electron microscope (SEM). Data were analyzed with 2-way ANOVA and pairwise comparison ( $\alpha=.05$ ).

**Results.** Significant interactions were found between aging time and ceramic material on  $L^*$ ,  $a^*$ ,  $b^*$ , and TP ( $P<.001$ ) as follows:  $b^*$  partial eta squared [ $\eta_p^2$ ]=0.689;  $L^*$   $\eta_p^2$ =0.186;  $a^*$   $\eta_p^2$ =0.176; and TP  $\eta_p^2$ =0.137. The  $b^*$  values significantly decreased after aging for zirconia ( $P<.001$ ), whereas TP increased after aging for zirconia ( $P<.014$ ) except at 10 hours ( $P=.389$ ) and for lithium disilicate ( $P<.001$ ). The  $\Delta E_{00}$  values relative to baseline ranged from 2.03 to 2.52 for aged zirconia and from 0.07 to 0.23 for aged lithium disilicate. XRD analysis revealed that hydrothermal aging promoted an increase in m-phase contents. AFM and SEM demonstrated surface alterations after aging.

**Conclusions.** Optical properties and microstructures of precolored monolithic zirconia ceramics were affected by hydrothermal aging, and translucency increased slightly with aging time. (*J Prosthet Dent* 2019;121:676-82)

depending on the amount of t→m phase transformation.<sup>16</sup> In addition, Y-TZP could be decreased in fatigue strength by about a factor of 2 to 3 under critical cyclic loading in the presence of water.<sup>17</sup> However, Cotes et al<sup>18</sup> reported that LTD did not affect the flexural strength of Y-TZP ceramics, although t→m phase transformations were induced by an autoclave aging process.

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## Clinical Implications

Some color changes may be evident in precolored monolithic zirconia restorations with intraoral use, and thus, an esthetically unacceptable mismatch might be expected approximately 3 to 4 years after delivery of the prostheses.

The clinical LTD of Y-TZP has been simulated by steam autoclave treatments at increased temperatures (120°C to 140°C) because the  $t \rightarrow m$  phase transformation is thermally activated and accelerated by the presence of water.<sup>9,12,16,19</sup> One hour of artificial aging treatment in an autoclave at 134°C has been reported to be equivalent to 3 to 4 years in vivo at 37°C.<sup>9</sup> Chevalier et al<sup>20,21</sup> designed accelerated aging in an autoclave at 134°C under 0.2 MPa over a period of 10 hours. In other studies,<sup>22,23</sup> the artificial aging protocol was performed in an autoclave at 134°C under 0.2 MPa for 20 hours.

For the long-term clinical success of esthetic restorations, the color should be stable to ensure reliable color matching. However, the color and translucency of dental ceramics is affected by hydrothermal aging.<sup>1,24-29</sup> Dental zirconia ceramics have been successively developed with improved translucency and esthetics similar to those of lithia-based glass-ceramics, making them suitable for use in the anterior region.<sup>30</sup> Precolored monolithic zirconia ceramics have been introduced with enhanced color reproduction. Although precolored anatomic contour zirconia restorations bring esthetic and biomechanical advantages, information on how their optical properties are affected by LTD is lacking. Therefore, the purpose of this in vitro study was to evaluate the effect of hydrothermal aging on the optical properties, phase transformation, and surface topography of precolored monolithic zirconia ceramics. In addition, the long-term color stability of precolored zirconia was compared with that of precolored lithium disilicate. The null hypothesis was that no significant differences would be found in the optical properties of zirconia specimens subjected to accelerated artificial aging with increasing aging time.

## MATERIAL AND METHODS

Fifty square-shaped precolored monolithic zirconia ceramic specimens (17.0×17.0×1.5 mm; ZrO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub> 7.12% to 7.16%, and so forth.; Katana ML A Light; Kuraray Noritake) and 50 IPS e.max CAD lithium disilicate glass-ceramic specimens (16.0×16.0×1.5 mm; SiO<sub>2</sub>, Other oxides; LT A2; Ivoclar Vivadent AG) as a reference group were prepared. The final thicknesses of the specimens were adjusted to 1.5 ±0.01 mm, which is the manufacturer-recommended occlusal wall thickness for posterior monolithic zirconia crowns, using a

horizontal grinding machine (HRG-150; AM Technology). Katana ML A Light (corresponding to Vita A 1.5 to 2 as specified by the manufacturer) is a precolored multilayered zirconia, and in an attempt to get specimens with the same color values, the specimens were cut from the lowermost layers of the blocks, which has been reported to exhibit higher chroma values than those of the upper part.<sup>31</sup>

Subsequently, the bottom surface of the specimen was polished by using a multistep protocol (coarse, medium-coarse, and super-fine grit; Edenta) followed by diamond paste (Legabril Diamond; Metalor Dental). All measurements were carried out on the polished surfaces of the specimens. The specimens were further heat-treated at 930°C with 1 hour of holding time, followed by slow cooling to 500°C in a furnace, and then cooled slowly in air which could lead to the  $m \rightarrow t$  phase transformation after annealing.<sup>32</sup> Before the test, the specimens were ultrasonically cleaned in an isopropyl alcohol bath for 5 minutes. The specimens were artificially aged in an autoclave (SAC-230G; Shinhung) at 134°C under 0.2 MPa and divided into 5 subgroups according to the aging time (n = 10), namely baseline (control), 1, 3, 5, and 10 hours.

The spectral reflectance was measured with a spectrophotometer (Color iControl; X-Rite) from 360 to 750 nm at 10-nm intervals against a white polytetrafluoroethylene background (GM29010330; X-Rite; Commission Internationale de l'Éclairage (CIE)  $L^*=93.886$ ,  $a^*=-0.008$ , and  $b^*=2.474$ ), a black glass-ceramic tile (CM-A101B; Konica Minolta; CIE  $L^*=0.11$ ,  $a^*=-0.014$ , and  $b^*=0.018$ ), and an A2 glass-ceramic tile (IPS e.max Press MO; Ivoclar Vivadent AG; CIE  $L^*=75.632$ ,  $a^*=-1.242$ , and  $b^*=11.232$ ). With a 6-mm diameter aperture and a 6-mm diameter measurement area, diffuse/8-degree geometry and specular component-excluded (de:8°) geometric condition were used. Five measurements per specimen were made, and so 50 data points were collected for each group. The reflectance data were then converted to color parameters according to the CIE Lab color space relative to D65 using the 2-degree standard observer (CIE, 1931). The mean color values against an A2 background were used for the color difference calculation. CIEDE2000 color differences ( $\Delta E_{00}$ )<sup>33,34</sup> between specimen pairs for Katana and e.max were calculated as:

$$\Delta E_{00} = \left[ \left( \frac{\Delta L'}{K_L S_L} \right)^2 + \left( \frac{\Delta C'}{K_C S_C} \right)^2 + \left( \frac{\Delta H'}{K_H S_H} \right)^2 + R_T \left( \frac{\Delta C'}{K_C S_C} \right) \left( \frac{\Delta H'}{K_H S_H} \right) \right]^{\frac{1}{2}}$$

The translucency parameters (TPs) of the specimens for each period were obtained by calculating the  $\Delta E_{00}$

between the values against white and black backgrounds.<sup>35</sup>

One representative Katana specimen from each aging group was subjected to crystalline phase analysis by X-ray diffraction (D8 ADVANCE; Bruker) using Cu-K $\alpha$  radiation ( $\lambda=1.5406$  Å) over the range 2 $\theta$  to 36 $\theta$  with a step size of 0.01 degrees at a scanning rate of 2 degrees per minute. The monoclinic peak intensity ratio ( $X_m$ ) on the specimen's surface was calculated according to the method of Garvie and Nicholson<sup>36</sup>:

$$X_m = \frac{I_m(\bar{1}11) + I_m(111)}{I_m(\bar{1}11) + I_m(111) + I_t(011)}$$

Monoclinic volume content ( $F_m$ ) was calculated using the method of Toraya et al<sup>37</sup>:  $F_m = \frac{1.311X_m}{1 + 0.311X_m}$ .

For zirconia, the microanalyses of surface topography and microstructure were performed using an atomic force microscope (AFM; XE-100; Park systems) and a scanning electron microscope (SEM; JSM-7401F; JEOL Ltd). For the AFM examination, one representative Katana specimen from each aging group was scanned in the noncontact mode (PPP-NCHR-50 probes, Force constant=42 N/m) with acquisition of 512 $\times$ 512 pixels per image (10 $\times$ 10  $\mu$ m) at a scan rate of 0.20 Hz using a specific software (XE-100; Park systems). The average arithmetic mean roughness value (Ra) was also determined by AFM. One representative Katana specimen from the baseline subgroup and one from 10 hours subgroup were thermally etched at 1400°C for 1 hour in air to reveal grain boundaries and then sputter coated with platinum (Q150T Sputter Coater; Quorum Technologies Ltd). SEM images were made at magnifications of  $\times$ 9000 and  $\times$ 15 000.

Statistical analyses were performed with statistical software (IBM SPSS Statistics, v23.0; IBM Corp). The Levene test and the Shapiro-Wilk test were conducted to check the assumption of homogeneity and normality for the dependent variables (CIE  $L^*$ ,  $a^*$ ,  $b^*$ , and TP) across all levels of the independent variables (aging time and ceramic material). Two-way ANOVA with the Tukey honestly significant difference multiple comparison test for post hoc analysis was used to determine how the outcomes were affected by the 2 factors. Simple main effects were performed to verify the degree to which one factor was differentially effective at each level of a second factor with the statistical software's command syntax. Correlation and regression analyses were carried out to assess the relationships between variables. The expected statistical powers with the chosen number of measurements (50 measurements per group) were 98.6% for 2-way ANOVA and 93.3% for Pearson correlation (G\*Power v3.1.9.2; Duesseldorf University) ( $\alpha=.05$  for all tests).

**Table 1.** Means  $\pm$ SD for CIE  $L^*$ ,  $a^*$ , and  $b^*$  values against an A2 background and TP values of each subgroup

Material	Aging Time	$L^*$	$a^*$	$b^*$	TP
Katana (monolithic zirconia)	Baseline	70.68 $\pm$ 0.78 <sup>a</sup>	0.91 $\pm$ 0.06 <sup>a</sup>	14.11 $\pm$ 0.26 <sup>c</sup>	4.81 $\pm$ 0.22 <sup>a</sup>
	1 h	71.86 $\pm$ 0.43 <sup>c</sup>	1.07 $\pm$ 0.03 <sup>d</sup>	10.51 $\pm$ 0.44 <sup>a*</sup>	4.93 $\pm$ 0.27 <sup>b</sup>
	3 h	72.00 $\pm$ 0.41 <sup>c</sup>	1.01 $\pm$ 0.05 <sup>b,c</sup>	10.60 $\pm$ 0.33 <sup>a***</sup>	4.95 $\pm$ 0.08 <sup>b</sup>
	5 h	71.44 $\pm$ 0.51 <sup>b</sup>	1.03 $\pm$ 0.03 <sup>c</sup>	11.10 $\pm$ 0.52 <sup>b</sup>	5.07 $\pm$ 0.16 <sup>c</sup>
	10 h	71.56 $\pm$ 0.63 <sup>b</sup>	0.99 $\pm$ 0.01 <sup>b</sup>	11.03 $\pm$ 0.52 <sup>b</sup>	4.88 $\pm$ 0.09 <sup>a,b</sup>
e.max (lithium disilicate)	Baseline	66.72 $\pm$ 0.68 <sup>A</sup>	0.05 $\pm$ 0.10 <sup>A</sup>	10.43 $\pm$ 0.50 <sup>A</sup>	7.95 $\pm$ 0.28 <sup>A</sup>
	1 h	66.99 $\pm$ 0.13 <sup>A</sup>	0.05 $\pm$ 0.06 <sup>A</sup>	10.44 $\pm$ 0.52 <sup>A*</sup>	8.14 $\pm$ 0.25 <sup>B</sup>
	3 h	66.62 $\pm$ 0.49 <sup>A,B</sup>	0.04 $\pm$ 0.09 <sup>A</sup>	10.48 $\pm$ 0.56 <sup>A***</sup>	8.24 $\pm$ 0.13 <sup>B</sup>
	5 h	66.98 $\pm$ 0.29 <sup>A,C</sup>	0.02 $\pm$ 0.07 <sup>A,B</sup>	10.31 $\pm$ 0.37 <sup>A</sup>	8.22 $\pm$ 0.18 <sup>B</sup>
	10 h	66.69 $\pm$ 0.29 <sup>A,B</sup>	-0.01 $\pm$ 0.07 <sup>B</sup>	10.45 $\pm$ 0.44 <sup>A</sup>	8.42 $\pm$ 0.06 <sup>C</sup>

SD, standard deviation; TP, translucency parameter. Means with same superscript letter in each column are not significantly different based on pairwise comparisons for simple main effects of aging time ( $P>.05$ ). Means with same superscript characters (\* and \*\*) in each column are not significantly different from each other based on pairwise comparisons for simple main effects of ceramic material ( $P>.05$ ).

## RESULTS

The Shapiro-Wilk test indicated that each dependent variable was normally distributed ( $P>.05$ ). Two-way ANOVA analyses indicated significant interactions between aging time and ceramic material for  $L^*$ ,  $a^*$ ,  $b^*$ , and TP ( $P<.001$ ). The largest combined effect of the factors was for  $b^*$  (partial eta squared [ $\eta_p^2$ ]=0.689), followed by  $L^*$  ( $\eta_p^2=0.186$ ),  $a^*$  ( $\eta_p^2=0.176$ ), and TP ( $\eta_p^2=0.137$ ). After significant interactions, the simple main effects were analyzed by pairwise comparisons using the Sidak adjustment for multiple comparisons. The  $L^*$  values of aged Katana were higher than those of baseline ( $P<.001$ ), whereas there were no significant differences in  $L^*$  values for aged e.max ( $P=.062$ , .102, .966, and 1.000). The  $a^*$  values of aged Katana were higher than those of baseline ( $P<.001$ ), whereas there were no significant differences in  $a^*$  values for aged e.max ( $P=.170$ , .999, and 1.000) except at 10 hours ( $P=.001$ ). The  $b^*$  values of aged Katana were higher than those of baseline ( $P<.001$ ), whereas there were no significant differences in  $b^*$  values for aged e.max ( $P=.879$ , 1.000, 1.000, and 1.000). The TP values increased ( $P<.014$ ) except 10 hours ( $P=.389$ ) for Katana, and TP values increased ( $P<.001$ ) for e.max as a function of aging time. Means (standard deviation) for CIE  $L^*$ ,  $a^*$ , and  $b^*$  values against an A2 background and TP values of each subgroup based on the pairwise comparisons for simple main effects are listed in Table 1.

Pearson correlation coefficients ( $r$ ) between aging time and each outcome were calculated, and linear regression analyses were conducted to quantify goodness of fit with coefficient of determinations ( $R^2$ ). For the Katana groups, there was a very weak positive correlation between  $L^*$  value and aging time ( $r=0.157$ ,  $R^2=0.025$ ,

**Table 2.** CIEDE2000 color differences ( $\Delta E_{00}$ ) between specimen pairs for Katana and e.max

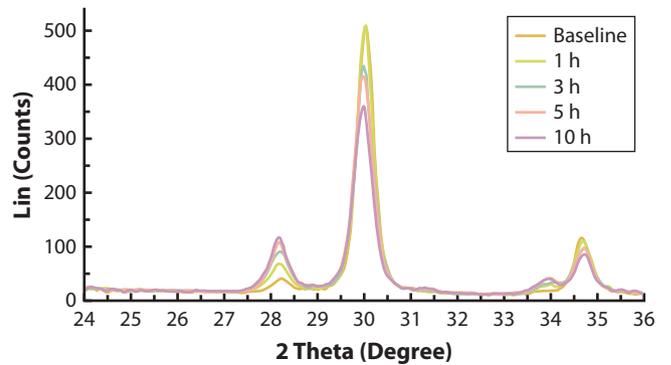
Specimen Pair	$\Delta E_{00}$	
	Katana	e.max
Baseline/1 h	2.52	0.22
Baseline/3 h	2.49	0.09
Baseline/5 h	2.03	0.23
Baseline/10 h	2.10	0.07
1 h/3 h	0.15	0.31
3 h/5 h	0.54	0.32
5 h/10 h	0.11	0.25

$P=.013$ ) and a weak negative correlation between  $b^*$  value and aging time ( $r=-0.394$ ,  $R^2=0.155$ ,  $P<.001$ ). The  $a^*$  and TP values increased slightly, but there were no statistically significant correlations ( $P=.059$  for  $a^*$  and  $P=.146$  for TP). For the e.max groups, there was a weak negative correlation between the  $a^*$  value and aging time ( $r=-0.253$ ,  $R^2=0.064$ ,  $P<.001$ ) and a moderate positive correlation between the TP value and aging time ( $r=0.574$ ,  $R^2=0.330$ ,  $P<.001$ ). No significant correlations were found between  $L^*$  value and aging time ( $P=.269$ ) or between  $b^*$  value and aging time ( $P=.890$ ).

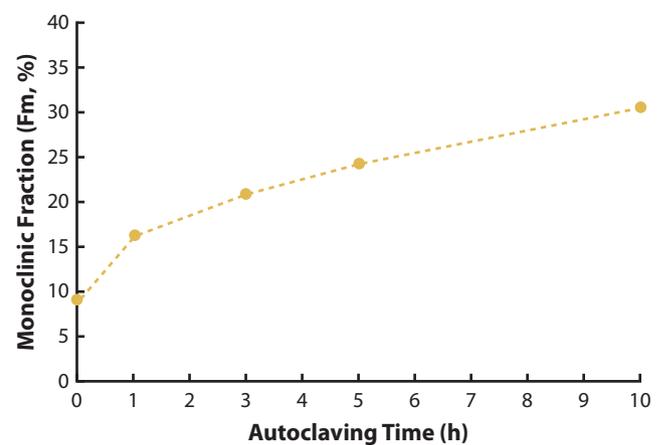
Table 2 shows  $\Delta E_{00}$  values between specimen pairs for Katana and e.max. For Katana groups,  $\Delta E_{00}$  values relative to baseline ranged from 2.03 to 2.52, which would be a clinically unacceptable color match based on the previous studies (1.80 to 2.25  $\Delta E_{00}$  units).<sup>38-41</sup> However,  $\Delta E_{00}$  values between 1 and 3 hours, between 3 and 5 hours, and between 5 and 10 hours were within the 50:50% perceptibility threshold (0.80 to 1.30  $\Delta E_{00}$  units).<sup>38-40</sup> However, for the e.max groups, the color differences relative to baseline ranged from 0.07 to 0.23  $\Delta E_{00}$  units and were within the 50:50% perceptibility threshold resulting in a stable color match.

The X-ray diffraction data for all subgroups of Katana are illustrated in Figure 1. Accelerated aging in an autoclave mainly caused an increase of m(111) peak which was the most stable m configuration<sup>42</sup> and, at the same time, triggered  $t \rightarrow m$  phase transformation leading to an increase of m(200) at 34.16 degrees ( $2\theta$ ). Figure 2 shows transformed monoclinic contents ( $F_m$ ) produced as a function of aging time. The initial monoclinic phase content of the control specimen was 9.51% and reached 30.79% after 10 hours of aging in an autoclave.

The representative AFM images of each subgroup of Katana are displayed in Figure 3. AFM analyses revealed the presence of parallel scratches created by polishing procedure, and increased monoclinic spots along these patterns were identified with increasing aging time. The Ra values were determined for each aging time. Surface roughness increased with aging time (Baseline: 8.12 nm, 1 hour: 8.99 nm, 3 hours: 10.04 nm, 5 hours: 10.12 nm, and 10 hours: 10.55 nm). SEM images of a control specimen and a specimen aged for 10 hours are shown in



**Figure 1.** X-ray diffraction patterns for all subgroups of Katana group at  $2\theta$  range between 24 to 36 degrees.



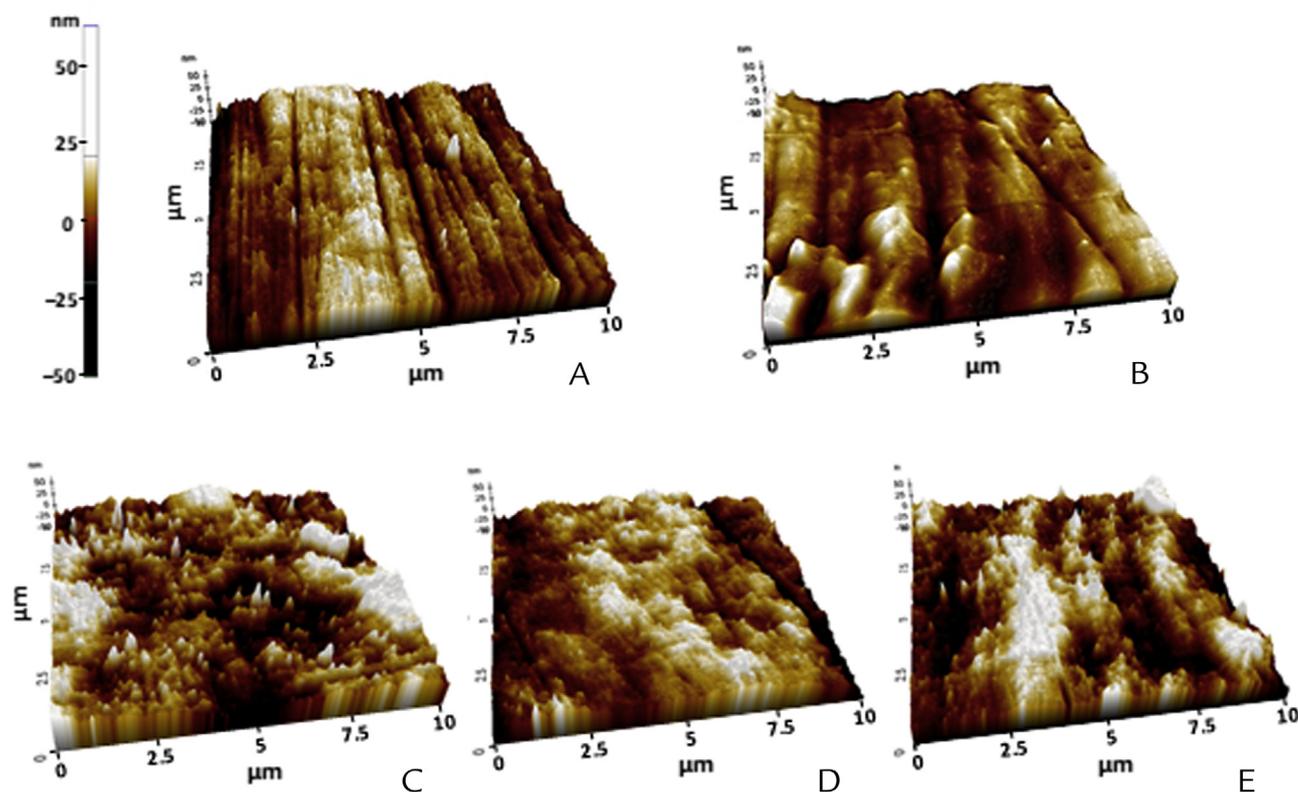
**Figure 2.** Monoclinic fraction ( $F_m$ ) obtained from the XRD data as function of aging time at 134°C/0.2 MPa. XRD, X-ray diffraction.

Figure 4. Alterations of topographic patterns induced by autoclave aging methods were detected, and the textured surface appearance was apparently observed on the specimen aged for 10 hours.

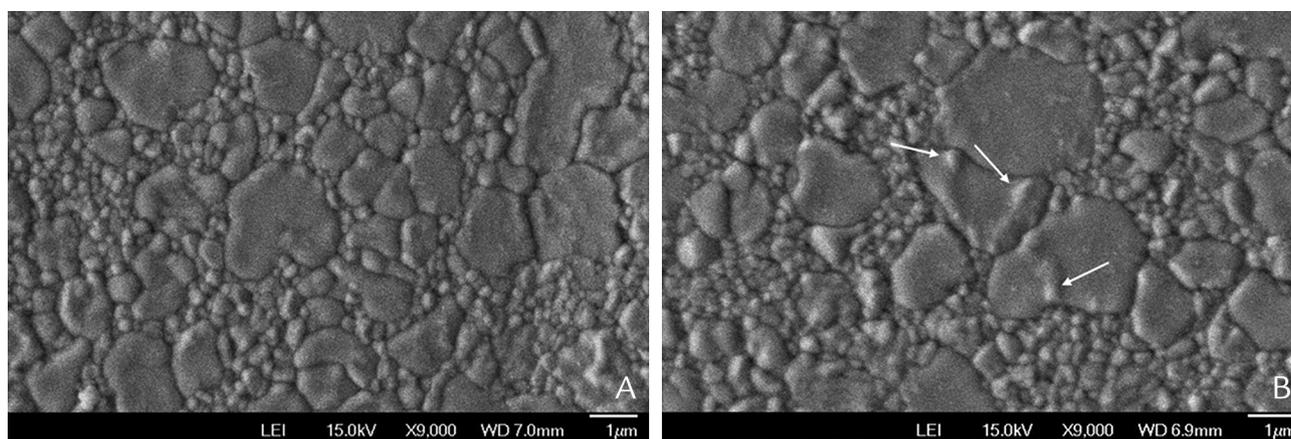
### DISCUSSION

According to the results of this study, the null hypothesis was rejected because hydrothermal aging significantly affected the optical properties of precolored dental monolithic zirconia ceramics. Noticeable changes in color parameters were observed as hydrothermal aging progressed, and a slight increase in translucency with increasing aging time except 10 hours was identified. Although weak correlations were found between color parameters and aging time, aged Y-TZP specimens displayed a brighter, redder, and more bluish appearance than nonaged Y-TZP specimens regardless of aging time.

Effects of LTD on the optical properties of Y-TZP have been studied. Fathy et al<sup>25</sup> reported that the formation of pores and the difference in refractive indices between monoclinic and tetragonal zirconia crystals would induce more light scattering, resulting in decreased translucency



**Figure 3.** Three-dimensional atomic force microscopy images ( $10\text{-}\mu\text{m}^2$  scan) for each subgroup of Katana group. A, Baseline; B, 1 h; C, 3 h; D, 5 h; E, 10 h.



**Figure 4.** Representative SEM photomicrographs (original magnification  $\times 9000$ ) of thermally etched Katana specimens. A, Baseline; B, 10 h. Arrows indicate transformation of grain surfaces with texture. SEM, scanning electron microscope.

after hydrothermal aging. Dikicier et al<sup>24</sup> showed that coloring agents could easily break down under UV radiation exposure, and as a result, the formation of peroxide caused the changes of color parameters. Nakamura et al<sup>19</sup> reported that  $\Delta E^*_{ab}$  values after 100 hours of autoclaving were within the perceptibility threshold even though color parameters of 3Y-TZP were affected by LTD. They also found that higher contents of trivalent dopants of coloring pigments ( $\text{Fe}_2\text{O}_3$  and  $\text{Er}_2\text{O}_3$ ) and higher cubic fractions in the colored 3Y-TZP led to

higher resistance to LTD than noncolored zirconia. Zhang et al<sup>1</sup> reported that dopant segregation of trivalent oxides ( $\text{Al}_2\text{O}_3$ ,  $\text{Sc}_2\text{O}_3$ ,  $\text{Nd}_2\text{O}_3$ , and  $\text{La}_2\text{O}_3$ ) to the grain boundaries induced hydrothermal stability and high translucency of anatomic contour 3Y-TZP. In addition, trivalent dopant promoted the adoption of cubic zirconia, and thus, optically isotropic cubic zirconia caused reduced birefringence at grain boundaries.<sup>27</sup>

In this work, although CIEDE2000 color difference was greater than 2.0 between baseline and 1 hour

( $\Delta E_{00}=2.52$ ), the difference was negligible thereafter for zirconia. In a similar way, Table 1 shows that changes in color values, especially a decrease in  $b^*$  values, occurred at 1 hour, and color values seemed to remain stable afterward for zirconia. However, the color parameters of lithium disilicate were relatively stable under aging treatments. As might be deduced from the results, larger color difference relative to baseline for zirconia than lithium disilicate mainly resulted from the changes in  $b^*$  values. Therefore, specific colorants might affect aging kinetics with regard to the behavior of oxygen vacancies in the first 3 to 4 years of intraoral aging. In addition, early thermal or electrochemical degradations of specific dye cations within Y-TZP ceramics may occur. Further relevant study is required.

In the present study, TP values increased with increasing aging time for both zirconia and lithium disilicate. Certain metal oxides such as coloring pigments might enhance the formation of cubic zirconia, resulting in higher TP values with increasing aging time because of the reduced light scattering from the grain boundaries of cubic zirconia. IPS e.max CAD, as the original version of lithium disilicate glass-ceramic, includes up to 4 wt%  $ZrO_2$  with additives for color and opalescence.<sup>30</sup> There could be any ionic interactions within the glass-ceramic crystalline phase leading to increased translucency after hydrothermal aging. For Y-TZP, a small decrease in the translucency at 10 hours was detected and explained perhaps by the increased light scattering caused by changes in surface morphology. Further research for a longer period of aging time is needed to verify the magnitude and direction of shifts in the translucency.

According to ISO standard 13 356:2008, the maximum amount of monoclinic phase of Y-TZP after autoclaving aging at 134°C under 0.2 MPa for a period of 5 hours should not exceed 25% for a suitable biomedical use.<sup>2</sup> In Pereira et al systematic review,<sup>3</sup> m-phase contents were about 0% to 14% before aging but increased to between 11% and 40% after accelerated aging for 10 hours. They also showed that flexural strength decreased only when more than 50% of m-phase was noted. In this study, increased t→m transformation behavior was found with increasing aging time, resulting in 24.37% of m-phase at 5 hours and 30.79% at 10 hours of artificial aging. Hence, the time needed to reach a 25% monoclinic fraction could be estimated at 20 years in the oral environment for precolored dental monolithic zirconia ceramics.

In the present study, polishing procedures were performed on the zirconia specimens and are recommended as an essential clinical procedure to enhance the outcome of the restorations.<sup>5</sup> Pereira et al<sup>6</sup> reported that a smoother surface could reduce the area for water interaction, resulting in lower susceptibility to hydrothermal aging. However, Cattani-Lorente et al<sup>5</sup> showed that mild

surface grinding increased aging resistance. Therefore, the presence of compressive stress due to surface treatments on the zirconia surface increased resistance against hydrothermal degradation.<sup>13</sup>

In this study, aging caused increased roughness of the zirconia specimens, which is consistent with the previous studies,<sup>5,7</sup> showing about a 30% increase in Ra value after artificial aging for 10 hours. Furthermore, previous studies<sup>5,7</sup> found that t→m transformation induced grain growth, which was associated with surface irregularities. Such irregular grain growth was also detected in AFM and SEM images of this study (Figs. 3 and 4). Volume expansion of approximately 4% induced by t→m phase transformations would lead to an increase in surface roughness, causing wear to the opposing dentition.<sup>8</sup>

In this study, hydrothermal treatment was applied to clarify the effects of aging on the optical properties of zirconia ceramics. This artificial aging technique can serve to predict the long-term degradation aging behavior of Y-TZP. However, in the oral environment, dental restorations will be exposed to different stimuli, including mechanical aging, thermocycling, and chemical aging. Therefore, to reflect the clinical situation, more studies are required to assess the changes in the optical properties of dental ceramics under various aging conditions. In addition, in the present study, aging effects on the optical properties of a single brand of higher translucency monolithic zirconia ceramic, but not indicated for use in esthetically demanding zones, were compared with those of a representative glass-ceramic. Further studies should evaluate aging effects on the optical properties of recently introduced esthetic dental ceramics with different compositions.

## CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Optical properties of precolored dental monolithic zirconia ceramics were affected by hydrothermal aging.
2. Translucency increased slightly with increasing aging time for Y-TZP and lithium disilicate glass-ceramics.
3. Increased phase transformations and surface alterations as a function of aging time were found on the surfaces of precolored monolithic zirconia ceramics.

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