

Effect of low-temperature degradation and sintering protocols on the color of monolithic zirconia crowns with different yttria contents

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This study investigated the effects of low-temperature degradation (LTD) on the L^* , a^* , and b^* values of highly translucent zirconia crowns. Four types of zirconia disks with different yttria contents (IPS e.max ZirCAD LT, IPS e.max ZirCAD MT, IPS e.max ZirCAD MT Multi, IPS e.max ZirCAD Prime, Ivoclar) and two shades (A2 and BL) were used. A crown was manufactured using four types of zirconia and LTD treated. Color measurements were performed, and the color difference (ΔE_{00}) before and after LTD was calculated. The microstructure was determined through X-ray fluorescence and X-ray diffractometry. Highly translucent zirconia crowns showed greater changes in the a^* and b^* values than in the L^* value after LTD, regardless of the shade. The Multi2 crowns exhibited a discernible color change due to the LTD treatment. The X-ray fluorescence results did not reveal any apparent change in the microstructure between sintering programs for all zirconia specimens.

Keywords: Aging, Esthetic dentistry, Optical property, Speed sintering, Y-TZP

INTRODUCTION

Recently, highly translucent zirconia has been developed for application to frameworks in dentistry; this zirconia has a higher translucency than conventional zirconia¹. Yttria-stabilized zirconia (YSZ) is currently used in dentistry, and there are three common types of zirconia according to the yttria content²: 3 mol% yttria-stabilized tetragonal zirconia polycrystals (Y-TZP), 4 mol% yttria-partially stabilized zirconia (4Y-PSZ), and 5Y-PSZ containing 3, 4, and 5 mol% yttria, respectively³. Highly translucent zirconia can be used to fabricate high-strength anatomical zirconia fixed partial dentures (FPDs) without the need for skilled porcelain layering, and has gained widespread clinical acceptance⁴. In addition, anatomical monolithic zirconia crowns have been reported to exhibit superior fracture resistance than anatomical glass-ceramic crowns⁵, as well as reduced wear on antagonist teeth^{6,7}. Monolithic restorations, as opposed to bilayered ones, offer distinct advantages, including less-abutment tooth preparation and no risk of porcelain fracture⁸. In addition, the integration of a digital workflow minimizes technical operations using optical impressions and computer-aided design and manufacturing (CAD/CAM) systems. Furthermore, the use of a speed sintering program enables the application of zirconia FPDs in a single visit^{9,10}.

However, monolithic zirconia restorations lack the

protective porcelain veneer and are directly exposed to the oral environment. Exposure of the zirconia surface in monolithic restorations to the oral environment causes low-temperature degradation (LTD) of the material^{11,12}. Furthermore, Y-TZP is susceptible to degradation after long-term use in humid and low-temperature environments, such as the oral cavity¹³. While conventional Y-TZP contains a small amount of alumina that can inhibit degradation¹⁴, translucent Y-TZP has a reduced alumina content, which increases its translucency¹⁵. Therefore, monolithic zirconia restorations may be more sensitive to LTD because they are not coated with veneering ceramic and contain exposed zirconia^{16,17}. Fathy *et al.* reported a higher susceptibility of monolithic zirconia materials toward LTD than other framework types of zirconia¹¹. In addition, long-term exposure to the oral environment adversely affects the esthetics of zirconia restorations due to LTD¹⁸. Furthermore, LTD strongly depends on the crystal phase and microstructures of the material and changes drastically with the yttria content^{13,19}. Therefore, translucent Y-TZP might be more susceptible to LTD than conventional Y-TZP.

The firing temperature is one of the most essential factors influencing the resistance to LTD. In addition, reducing the sintering time can accelerate the manufacturing process of zirconia FPDs clinically²⁰. Multilayered zirconia was developed to reproduce the

optical characteristics of natural teeth, and various translucent zirconia materials have been used clinically; however, the effects of zirconia-specific LTD on color changes remain unclear. Thus far, most studies on translucent zirconia have focused on the transparency and strength of zirconia with a reduced sintering time^{9,20-23}).

Therefore, in this study, we investigated the effects of LTD on the L^* , a^* , and b^* values of highly translucent zirconia crowns. Crown models were fabricated using different zirconia materials with varying shades, and conventional and speed sintering were performed on the specimens. The color differences in the monolithic zirconia crowns before and after LTD were compared and analyzed.

The null hypothesis for this *in vitro* study posited that there would be no significant changes in L^* , a^* , and b^* values due to LTD, regardless of the yttria content or sintering program.

MATERIALS AND METHODS

An abutment tooth model of the maxillary left central incisor (A55A-211, Nissin, Kyoto, Japan) was used. The abutment tooth was modified using diamond points (102R, 106RD, SF106RD, Shofu, Kyoto, Japan) to create a deep chamfer finishing line of approximately 0.8 mm. Four types of shade-gradient zirconia disks with different yttria contents (IPS e.max ZirCAD LT, IPS e.max ZirCAD MT, IPS e.max ZirCAD MT Multi, IPS

e.max ZirCAD Prime, Ivoclar, Schaan, Liechtenstein) and two types of shades (A2 and BL) were used (Table 1). The modified abutment tooth was fixed to the maxillary model, and the maxillary dentition was scanned using a desktop scanner (Shofu S-WAVE scanner D900, Shofu). Based on the scanned data, the crowns were designed using a dental CAD suite (GO2dental, Shofu). The crown design mirrored the contralateral homonymous teeth. The cement spacer was set at 50 μm , the incisal thickness of the crown was 3.0 mm, and the cervical and middle thicknesses were 0.8 mm. After the crowns were milled, conventional and speed sintering were performed in a sintering furnace (Programat S1 1600, Ivoclar) according to the sintering schedule specified by the manufacturer ($n=9$) (Table 2). After the crowns were sintered, they were not polished to avoid changing their surface textures.

LTD treatment was implemented for 5 h at 134°C and 0.2 MPa in an autoclave (autoclave PC-242HS, Hirayama, Saitama, Japan), in accordance with the accelerated aging test standard ISO 13356:2015²⁴. The specimens were ultrasonically cleaned using distilled water for 10 min and then air-dried. After LTD, each fabricated crown was placed in a dedicated dark box, following which the L^* , a^* , and b^* values of the crowns were measured using a non-contact dental spectrophotometer (Crystaleye, Olympus, Tokyo, Japan) at three locations on the labial side (cervical, middle, and incisal areas). Calibration was performed prior to each measurement according to the manufacturer's

Table 1 Shade-gradient zirconia materials used

		Zirconia materials		Manufacturer	Lot No.
Mono-composition	Mono1	IPS e.max ZirCAD LT	3Y-TZP	Ivoclar	A2: X30012 BL: W44335
	Mono2	IPS e.max ZirCAD MT	4Y-PSZ	Ivoclar	A2: X20291 BL: Y24368
Multiple-composition	Multi1	IPS e.max ZirCAD MT Multi	Cervical side: 4Y-PSZ Middle position: 4Y-PSZ, 5Y-PSZ Incisal side: 5Y-PSZ	Ivoclar	A2: X46116 BL: X28520
	Multi2	IPS e.max ZirCAD Prime	Cervical side: 3Y-TZP Middle position: 3Y-TZP, 5Y-PSZ Incisal side: 5Y-PSZ	Ivoclar	A2: Y24810 BL: Y14099

* Manufacturer's published values

Table 2 Sintering programs for each material

Conventional sintering		Sintering temperature	Holding time	Total sintering time
		1,500°C	120 min	9 h 50 min
Speed sintering	Mono1	1,530°C	60 min	2 h 55 min
	Mono2			2 h 30 min
	Multi1			4 h 25 min
	Multi2			2 h 26 min

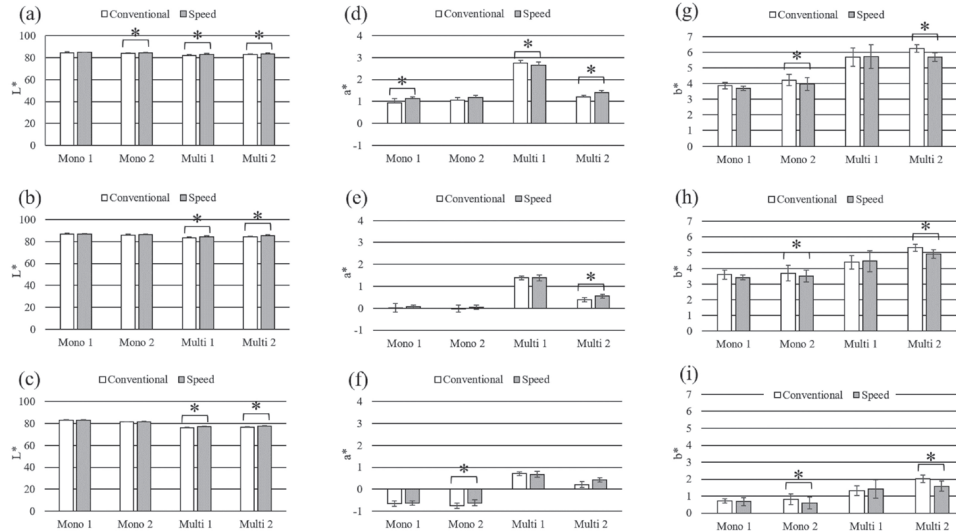


Fig. 3 Mean values (error bars=standard deviations) of L^* , a^* , and b^* as observed in the BL shade zirconia crowns ($n=9$). Mono1: mono-composition zirconia with 3 mol% yttria (3Y-TZP), Mono2: mono-composition zirconia with 4 mol% yttria (4Y-PSZ), Multi1: multiple-composition zirconia with 4 and 5 mol% yttria (4Y, 5Y-PSZ), and Multi2: multiple-composition zirconia with 3 and 5 mol% yttria (3Y-TZP, 5Y-PSZ). (a)–(c): L^* values, (d)–(f): a^* values, (g)–(i): b^* values. (a), (d), (g): cervical side, (b), (e), (h): middle, (c), (f), (i): incisal side

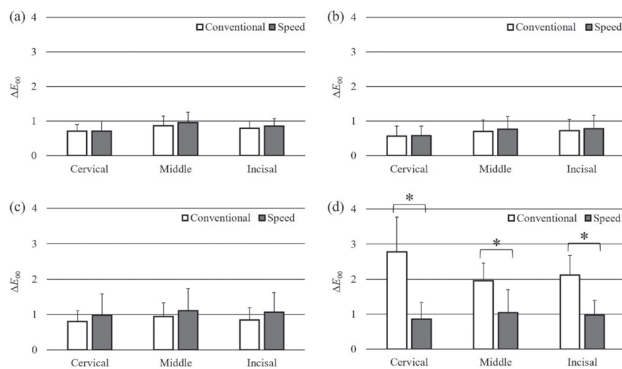


Fig. 4 Mean values (error bars = standard deviations) of the color difference (ΔE_{00}) between before LTD and after LTD in the A2 shade group ($n=9$). (a): Mono1 (3Y-TZP), (b): Mono2 (4Y-PSZ), (c): Multi1 (4Y-, 5Y-PSZ), (d): Multi2 (3Y-TZP, 5Y-PSZ). The data before LTD were taken from study by Miura *et al.*¹⁰

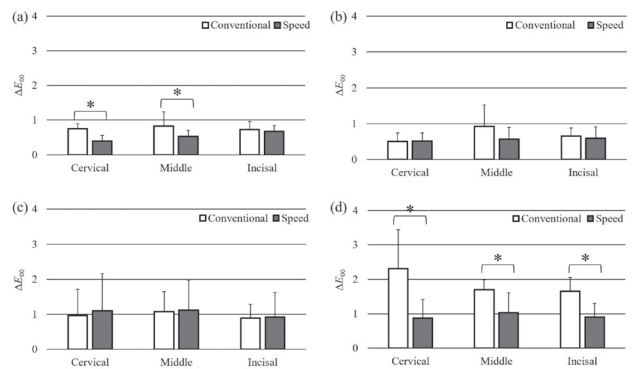


Fig. 5 Mean values (error bars=standard deviations) of the color difference (ΔE_{00}) between before LTD and after LTD in the BL shade group ($n=9$). (a): Mono1 (3Y-TZP), (b): Mono2 (4Y-PSZ), (c): Multi1 (4Y-, 5Y-PSZ), (d): Multi2 (3Y-TZP, 5Y-PSZ). The data before LTD were taken from study by Miura *et al.*¹⁰.

respectively. For the A2 and BL shades, the L^* values of Mono1 exhibited no significant differences between the conventional and speed sintering programs ($p>0.05$). Regardless of the difference in the measurement locations ($p>0.05$), there was no significant difference in the a^* value between the conventional and speed sintering programs for the A2 shade, Mono2 (Fig. 2). Similarly, regardless of the difference in the measurement locations ($p>0.05$), there was no significant difference in the b^* values between the conventional and speed sintering programs for BL shades Mono1 and Multi1 (Fig. 3). ΔE_{00} before and after LTD showed significant differences

at all measured locations between the conventional and speed-sintered Multi2 for the A2 and BL shades ($p<0.05$) (Figs. 4, 5). However, a significant difference in ΔE_{00} before and after LTD was observed for the BL shade, Mono1, between the cervical and middle locations ($p<0.05$) (Fig. 5).

The elemental compositions are shown as representative values (Table 3). The elemental compositions of Zr, Y, and Hf were almost identical for the conventional and speed sintering programs within the same material.

The XRD patterns of Multi2 specimens are shown

Table 3 Elemental composition analysis by X-ray fluorescence (%)

	Zr	Y	Hf	Others
Mono1-C	91.1	6.1	2.1	0.7
Mono1-S	90.5	6.1	2.3	1.1
Mono2-C	87.9	8.3	2.2	1.6
Mono2-S	88.0	8.3	2.3	1.4
Multi1-C	88.1	9.4	2.1	0.4
Multi1-S	87.6	9.0	2.2	1.2
Multi2-C	88.3	8.8	2.2	0.7
Multi2-S	88.2	8.6	2.2	1.0

C: conventional sintering

S: speed sintering

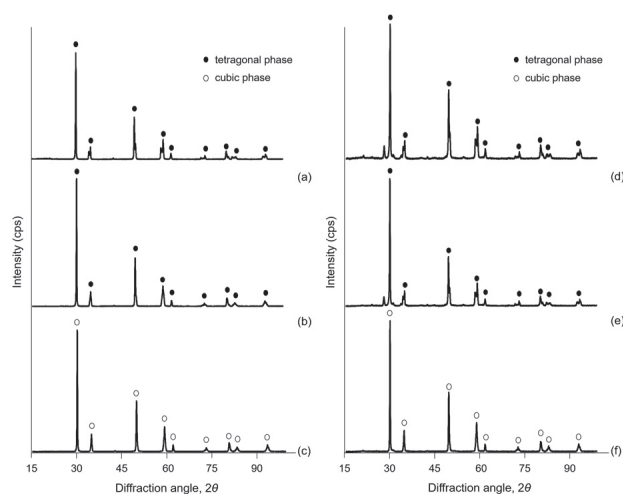


Fig. 6 XRD pattern of Multi2 (3Y-TZP, 5Y-PSZ) specimens of conventional sintering program.

(a)–(c): before LTD, (d)–(f): after LTD, (a)(d): cervical layer, (b)(e): middle, (c)(f): incisal layer. The data before LTD were taken from study by Miura *et al.*¹⁰.

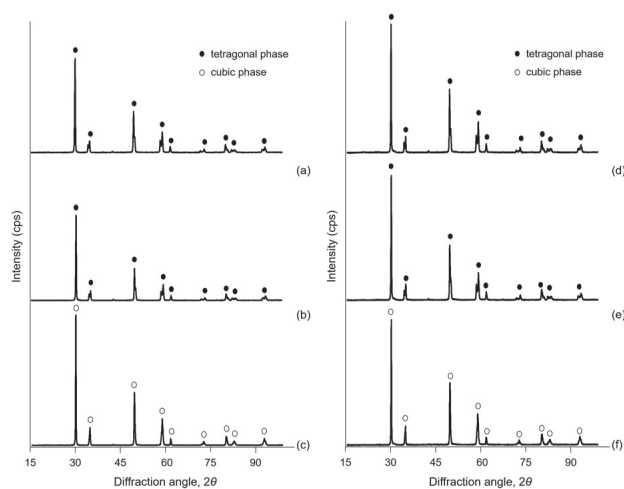


Fig. 7 XRD pattern of Multi2 (3Y-TZP, 5Y-PSZ) specimens of speed sintering program.

(a)–(c): before LTD, (d)–(f): after LTD, (a)(d): cervical layer, (b)(e): middle, (c)(f): incisal layer. The data before LTD were taken from study by Miura *et al.*¹⁰.

in Figs. 6 and 7. Notably, the XRD patterns before and after LTD of the specimens prepared by conventional and speed sintering are similar. For each before and after LTD specimens, the characteristic reflections were observed between 30° and 31° in 2θ. In the incisal layer of the Multi2 specimen, the cubic phase was observed because this specimen is 5Y-PSZ, which has a high Y₂O₃ content (Figs. 6, 7). No difference in crystal phase was observed in the Multi2 specimens before and after LTD.

DISCUSSION

In this study, the effect of LTD on the color of monolithic zirconia crowns fabricated from mono- and multi-composition shade-gradient zirconia using different

sintering programs was evaluated. It has been reported that the LTD of zirconia with 1 h of autoclave treatment at 134°C and 0.2 MPa has a theoretically equivalent effect of 3–4 years *in vivo*²⁶. Therefore, the 5 h of LTD processing time in this study could be considered equivalent to 15–20 years *in vivo*. However, for 3Y-TZP, it has been also reported that a 1-h hydrothermal treatment at 134°C for 3Y-TZP without alumina is equivalent to about 0.5 years of aging at 37°C, whereas for 3Y-TZP with alumina, it is equivalent to 2.5 to 3.5 years of aging at 37°C²⁷.

The null hypothesis that no difference would be found in the color of zirconia with different yttria contents through LTD treatment was not rejected, except for the *L** value of Mono1. For the *L** values of Mono1

crowns of mono-composition zirconia with 3 mol% yttria, the A2 and BL shades did not show any significant differences at any measured locations. For the Multi2 crowns of multiple-composition zirconia with 3 and 5 mol% yttria that were treated using the conventional sintering program, the color difference before and after LTD treatment resulted in $\Delta E_{00} > 1.8$ for all measured locations for A2 shade and at the cervical location for BL shade.

The CIEDE2000 color difference (ΔE_{00}) formula was used to calculate the color, lightness (L), chroma (C), and hue (H) differences in this study. Recent studies have suggested that this new formula can be used in dental research and applications^{18,25,28}. It has been reported that using the CIEDE2000 color difference formula yields a better fit than the previously used CIELAB (ΔE_{ab}) formula and improves the correlation between the visual color and calculated differences^{29,30}. Generally, it is impossible for the human eye to differentiate between minimal numerical color differences in dental materials. According to previous studies on porcelain color changes, ΔE_{ab} values lower than 1 are not clinically detectable by the human eye³¹. Furthermore, the CIELAB 50:50% perception threshold (PT) and 50:50% acceptance threshold (AT) in dentistry are $\Delta E_{ab} = 1.2$ and $\Delta E_{ab} = 2.7$, respectively, and the corresponding CIEDE2000 values are $\Delta E_{00} = 0.8$ and $\Delta E_{00} = 1.8$, respectively³². An excellent color match could be achieved when $\Delta E_{00} \leq 0.8$, and an acceptable color match could be obtained when $0.8 < \Delta E_{00} \leq 1.8$ ³³. Therefore, in this study, $\Delta E_{00} = 1.8$ was used as an index of color difference.

Park observed no changes in the L^* , a^* , and b^* values of shade-gradient zirconia with 5 mol% yttria after LTD treatment; however, the color difference was the highest between the incisal and middle layers in the layered zirconia ($\Delta E_{ab} = 7.11$). This value was a clinically unacceptable color difference. They stated that the color difference was primarily attributed to the difference in chroma³⁴. Conversely, Volpato *et al.* evaluated the color stability of zirconia with 3 mol% yttria after LTD treatment and found that the color difference ($\Delta E_{00} = 0.79\text{--}0.95$) increased with increasing LTD treatment time¹⁸. However, they could not confirm any changes on the zirconia surface due to aging after 4 h of LTD treatment¹⁸. Previous reports have suggested that extended LTD treatment and a shorter sintering time may affect the surface structure of zirconia or cause color changes^{20,34}. Although color differences were observed in the Multi2 crowns before and after LTD treatment, all LTD treatments were performed under constant conditions, and it remains to be examined whether the speed sintering program caused changes in the microstructure of the zirconia surface. Furthermore, it has been reported that LTD is related to the surface quality of the crown¹⁹. In this study, no polishing was performed to modify the material conditions. However, studies have reported variations in the L^* and a^* values depending on whether or not the zirconia surface is polished, and have reported that ΔE_{ab} is 2.7 or higher³⁵. Consequently, even after LTD, there are differences

in the L^* and a^* values depending on whether or not polishing is performed, possibly increasing ΔE_{ab} .

Dikicier *et al.* studied the effect of the core frame thickness and LTD treatment on the color difference of several ceramic materials (glass-infiltrated aluminum oxide, lithium disilicate glass ceramics, and 3Y-TZP)³⁶. The aging procedure involved exposing the ceramic specimens to UV light and water-spraying them for 200 h in a weathering machine. Their study reported that 300 h of artificial aging is equivalent to one year of clinical service. They reported that LTD decreased the L^* and b^* values and increased the a^* values for all zirconia specimens with three different thicknesses (0.5, 0.8, and 1.0 mm). A color difference ($\Delta E_{ab} = 1.06\text{--}1.29$) was observed in Y-TZP after aging; however, the value was lower than those of the glass-infiltrated alumina and lithium disilicate glass ceramics³⁶. A comparison of ΔE_{ab} before and after LTD due to the difference in thickness revealed that ΔE_{ab} was the largest at 0.5 mm; however, it was reported that there was no statistically significant difference in the relationship between the zirconia thickness and LTD³⁶. The specimens appeared more blue/red because of increasing a^* and decreasing b^* values³⁶. Although the aging method and clinical equivalent time of artificial aging were different, the increase in the a^* value was the same as in this study. In addition, this value is much lower than the ΔE_{ab} value ($\Delta E_{ab} = 5.03$) reported by Kurt and Bal³⁷ for the UV light aging method. However, this difference is attributed to the difference in the yttria and alumina contents between the zirconia in the core frame and the monolithic zirconia materials.

Other researchers have reported that color changes in veneering ceramic materials upon aging might be related to the metal oxide content^{36,38,39}. Metallic pigments are added to deepen the color of veneering ceramics, and these oxides are easily broken down by UV light. Consequently, the significant color change in the zirconia material could be attributed to the dissolution of metal oxides induced by UV light during aging³⁷. It has been reported that the use of metal oxides to obtain various shades in 3Y-TZP dental restorations could lead to crystallographic and microstructural changes that could affect their mechanical properties⁴⁰. Er, Pr, and Tb effectively provide natural tooth-like fluorescence. The zirconia A2 shade used in this study contained Er, Pr, and Tb, whereas the BL shade contained Er¹⁰. However, the pre-shaded zirconia used in this study was mixed with a predetermined amount of colored zirconia powder at the cold isostatic pressing and firing stages, and it is considered that the addition of Er, Pr, and Tb had no effect on the physical properties and color.

In this study, the XRF results for conventional and speed sintering showed no significant difference in the elemental compositions. In previous reports on the XRF of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ, the primary elements were Zr, Y, and HF, and for Zr, the content concentration was almost the same as in the present study⁴¹. In addition, it was reported that 3Y-TZP did not show any change, as deduced by comparing the XRF results before

and after LTD treatment⁴²).

In the present study, the ΔE_{00} values before and after LTD were greater in zirconia with multiple compositions. The changes in the a^* and b^* values due to LTD processing affected ΔE_{00} . This phenomenon was prominent in zirconia containing 3 mol% yttria, and was particularly noticeable in zirconia with multiple compositions. Although the translucency parameters (TPs) were not measured in this study, the TP of mono-composition 5Y-PSZ zirconia (Katana STML) was reported to be unaffected by conventional and speed sintering programs⁴³. Additionally, 60 h of LTD significantly reduced the TP values of the tested 3Y-TZP conventional zirconia (CEREC Zr) and speed-sintered zirconia (inCoris TZI). However, 5Y-PSZ zirconia (Katana STML), obtained by conventional and speed sintering, was reported to have no effect on the TP value⁴³. On the other hand, Kanpalta *et al.* reported that LTD affects the translucency of monolithic zirconia materials. It also reported that phase transformation to monoclinic phase was observed in mono-composition 6Y-PSZ zirconia (Katana UTML), however, not in PSZ with low yttria content⁴⁴. Furthermore, the similarity of the XRD patterns before and after LTD was similar to the present study. Although a color difference before and after LTD was observed in Multi2, no change in the XRD pattern or crystal phase was observed from the XRD results. These results suggest that the changes in L^* , a^* , and b^* due to LTD are due to 3 mol% yttria. Ban reported that zirconia dental prostheses could withstand decades of use in the oral cavity. It was reported that the LTD of zirconia in a 134°C autoclave represented an accelerated test of degradation phenomena near oral temperatures, and that it was inappropriate to point to the LTD of zirconia as a drawback of dental zirconia¹⁹.

Light transmission or translucency is one of the essential optical properties of all-ceramic restorations, and is affected by the background color. Although all-ceramic crowns are sometimes suggested for endodontically treated and discolored teeth in clinical practice, it is clear that the masking ability of the crown material affects the final esthetic results⁴⁵. Therefore, the monolithic zirconia material is affected by the abutment tooth color, resulting in a color difference from the final color. As some monolithic zirconia materials are affected in terms of color due to sintering conditions and LTD, the clinician needs to make appropriate choices when faced with various esthetic challenges. This study was limited by the use of only four types of monolithic zirconia crowns, two shades, and conventional and speed sintering programs to examine color changes under the influence of LTD. Various types of zirconia with different multilayered structures and yttria contents exist, and different sintering programs might cause color changes. Additionally, multiple factors, such as the crown thickness and abutment tooth color, can affect the final color of a monolithic zirconia crown, which is essential to match the natural tooth color accurately. Therefore, these multifaceted factors must be considered when selecting zirconia materials and shades. Currently,

clinical studies on the influence of sintering parameters and LTD treatment on the prognosis of monolithic zirconia restorations are lacking, and further research is encouraged in this endeavor.

CONCLUSION

Within the limitations of the present study, the L^* , a^* , b^* , and ΔE_{00} values of four types of zirconia were affected by LTD treatment. Highly translucent zirconia crowns with different yttria contents showed greater changes in the a^* and b^* values than in the L^* value after LTD, regardless of the shade. For Multi2 crowns with 3 and 5 mol% yttria, the LTD treatment caused a discernible color change.

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