Effect of Accelerated Aging on Color Stability of Two Silica-Based Ceramics with Leucite and Lithium-Disilicate Crystalline Phases After Glazing and Polishing

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Abstract

Background and Aim: Color match between prosthetic restorations and natural teeth is challenging in dentistry. The aim of the current study was to assess the effect of accelerated aging on color stability of two silica-based ceramics with leucite and lithium disilicate crystalline phases after glazing and polishing surface treatments.

Materials and Methods: In this experimental study, 30 disc-shaped specimens (1.5x10mm) of IPS e.max Press and Ceramco III ceramics were fabricated according to the manufacturers’ instructions. The specimens were randomly divided into two groups of surface treatments (glazing and polishing) and were then subjected to accelerated aging for 300 hours. Color parameters were measured before and after aging using a spectrophotometer. The L*, a* and b* parameters and the color change (∆E) of ceramics (based on the CIE L*a*b* system) before and after glazing were determined by spectrophotometry. Data were analyzed using two-way ANOVA and t-test. P<0.05 was considered statistically significant.

Results: After 300 hours of aging, the color stability (∆E), ∆L and ∆b of polished group was less than that of glazed group. The two ceramic types were not significantly different in terms of ∆E (range: 1.31 to 2.53; P=0.055).

Conclusion: Glazed silica-based ceramics had higher color stability than polished ceramics after aging in our study. Color stability of silica-based ceramic systems was influenced by the surface texture and not the type of crystalline phase.

Key Words: Lithia Disilicate, Aging, Discoloration

Introduction

Color match between prosthetic restorations and natural teeth is challenging in dentistry. Achieving a vital appearance by dental ceramics is difficult and high technical sensitivity. In natural teeth, irradiated light is reflected, emitted and absorbed by the enamel and dentin. Enamel crystals are responsible for light emission while dentin is primarily responsible for light absorption [1,2]. Light emission and specular reflection occur at the surface and are influenced by the surface texture of teeth. In dental ceramic application, the goal is to...
mimic the tooth color and create a natural appearance by a combination of light absorption and emission [3]. Thus, dental ceramics must possess the translucency of enamel and opacity of dentin in order to resemble the structure of natural teeth [4]. There is no ceramic product with both opacity and translucency. Therefore, the manufacturers have produced different types of ceramics for the core, opaque dentin ceramics, glazed andlucent veneering ceramics for application on the surface using the layering technique [5]. Feldspathic dental ceramics have a favorable behavior in light irradiation and are well capable of mimicking the natural tooth color. Feldspathic porcelains are esthetically favorable but have low strength. New ceramic systems have relatively opaque core for higher strength. Many of these systems require the veneering porcelain to provide optimal esthetics but the core participates in the color and translucency of the final restoration [6]. Considering the significance of esthetics in today’s world, color stability ofesthetic dental restorative materials is critically important. Optimal restorative materials must have high esthetics as well as durable and stable mechanical properties [7].

Data regarding the discoloration of ceramics over time are limited. Accelerated aging simulates the oral clinical setting for restorative materials and allows for thorough evaluation of color change of ceramics in the oral environment over time [8]. The Munsell and CIE L*a*b* color systems are used to describe color parameters. The parameters used in Munsell system include value, hue and chroma while the CIE L*a*b* system is based on three parameters of x, y and z, determined based on spectral reflection. The color change in this system is calculated by the square root of the sum of squares of change in L*, a* and b* parameters and is referred to as ∆E [9].

A glazed ceramic surface has several advantages. Glazing fills the gaps present on the baked porcelain surface and further increases its fracture strength. Also, it lowers the wear potential of the ceramic surface [10]. The efficacy of finishing and polishing procedures to obtain a smooth ceramic surface comparable to that of glazed ceramics has been extensively evaluated. Previous studies have evaluated the glazed ceramic surfaces by a profilometer, scanning electron microscopy (SEM) or visual inspection and recommended the application of finishing and polishing procedures as an alternative to glazing for porcelain restorations [11-15]. Studies on the mechanical properties of ceramics are limited and scarce information exists on the effects of polishing and glazing on the color stability of ceramic systems after accelerated aging. According to the literature, the aim of the current study was to assess the effect of accelerated aging on the color stability of two silica-based ceramics with leucite and lithium disilicate crystalline phases after glazing and polishing surface treatments.

**Materials and Methods**

This in vitro experimental study was conducted on 30 disc-shaped specimens (1.5x10mm) of IPS e.max Press and Ceramco III ceramics fabricated according to the manufacturers’ instructions. A Teflon mold measuring 10mm in diameter and 1.5mm in thickness was used for fabrication of the discs. The materials were IPS e-max Press core (Lot# p22617; Ivoclar Vivadent, Schaan, Liechtenstein), IPS e-max Ceram veneering (Lot# p22617; Ivoclar Vivadent, Schaan, Liechtenstein) and Ceramco III (Lot#03120604; Dentsply, Sirona, USA). The crystalline phase was lithium disilicate, fluorapatite and leucite. The composition of the materials was 70-60% of 0.5-4 µm second phase lithium ortho-phosphate, 0.1-0.3 µm, 23-19% of 300 µm fluorapatite and 1-5 µm leucite, respectively.

**Fabrication of lithium disilicate-based ceramic discs:**

Discs had a lithium disilicate core and fluorapatite veneering and were fabricated by the heat-press technique. To fabricate the core, wax pattern with 0.8mm thickness and 1cm diameter was first fabricated using a split mold and flasked with OPS Press Vest dental stone. Wax burnout was performed at 850°C for 20 minutes. High translucency ingots (A2, shade; IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) were placed in the flask and pressed using a plunger. The specimens were placed in the IPS ceramic furnace (EP500 Combi; Ivoclar Vivadent AG, Schaan, Liechtenstein) with a starting temperature of 700°C. The temperature was raised to 920°C.
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within 22 minutes. Injection was continued for 4 to 5 minutes. After cooling, the cores were removed by cutting the cylinder and sandblasting and were then immersed in acid (Invex) for 20 to 30 minutes for cleaning. The A2 shade of ceramic powder (e.max Ceram) was then applied in 0.7mm thickness and heated in a furnace. The veneering layer was baked at 750°C. This process took 8 minutes. The entire working time (including the cooling time) was 15 minutes. The specimens were then finished using diamond burs (Noritake Co., Tokyo, Japan) to standardize their thicknesses. A digital caliper with 0.01mm accuracy was used for the measurement of specimen thickness.

Fabrication of leucite-based ceramic discs:
Ceramco III discs (Dentsply, Sirona, USA) were fabricated by sintering. Ceramic powder (A2 body shade) was mixed with liquid and packed in a Teflon mold (10mm in length and 1.5mm in thickness). The excess water was absorbed by a paper towel under vibration. The specimens were then baked in a ceramic furnace (Vacumat 200, Vita Zahnfabrik, Germany) at 940°C.

The specimens in each group were randomly divided into the following subgroups:

- **Finishing and polishing subgroup**: Noritake finishing and polishing kit (Noritake Co., Tokyo, Japan) was used for finishing and polishing of ceramic discs. Ceramic discs were finished and polished with wet 240-1200 grit silicon carbide abrasive papers (Meister cones yellow-green) followed by the use of ceramic polishing paste (Noritake Pearl surface). Polishing time was 10 seconds for each step. All rotary instruments were used in a low-speed hand piece operating at 10,000 rpm under water coolant according to the manufacturers’ instructions.

- **Glazing subgroup**: The Ceramco III and IPS e.max Press specimens were glazed for one minute at 930°C and 740°C, respectively according to their manufacturers’ instructions. The specimens were then cleaned in distilled water in an ultrasonic bath (sw 1500, Citizen, Tokyo, Japan) and subjected to accelerated aging.

Accelerated aging:
All specimens were subjected to accelerated aging (water spray and UV irradiation) for 300 hours (weather-o-meter; Atlas Electric Devices, IL, USA). The irradiation rate by xenon lamp was 0.55 W/m². A 4500W xenon lamp was used to simulate day light (D65). The back panel temperature was between 38°C (dark) and 70°C (light) with a relative humidity between 95% (dark) and 50% (light). The temperature of dry bulb was 38°C (dark) and 47°C (light). The test cycle included 40 minutes of light along with water spray, 60 minutes of light alone and 60 minutes of dark along with water spray. The entire exposed energy was 389.2 kJ/m. The accelerated aging parameters were chosen based on previous studies [16,17] and 300 hours of aging under these conditions corresponded to one year of clinical service. After aging, the samples were subjected to measurement of color parameters by spectrophotometry. To assess the effect of polishing and glazing on the ceramic surface in microscopic scale, one specimen of each group was randomly selected and mounted on a metal stub and gold sputter coated for evaluation under field emission SEM at x500 magnification (S-4160; Hitachi, Tokyo, Japan). The L*, a* and b* parameters of the discs in different groups were determined and reported using spectrophotometry. The color change in each group (compared to the reference specimen) was calculated using the formula below:

\[
\Delta E(L^*a^*b^*) = \sqrt{(L_1^* - L_2^*)^2 + (a_1^* - a_2^*)^2 + (b_1^* - b_2^*)^2}
\]

Data were analyzed using SPSS version 11.5 (SPSS Inc., IL, USA). Color parameters of the groups with different surface treatments were
Results

The mean and standard deviation of $L^*$ value in ceramic specimens with two different surface treatments before and after aging are shown in Table 1. The baseline $L_b^*$ (before aging) in Ceramco III specimens was significantly higher than that of e-max Press specimens ($P<0.0001$); however, no significant difference was found between glazed and polished specimens ($P=0.181$). The interaction effect of ceramic type and type of surface treatment on this color parameter was not significant ($P=0.934$).

The $\Delta L$ (difference between $L_a^*$ after aging and baseline $L_b^*$) was negative for both ceramic types. It means that the $L_a^*$ parameter decreased after aging (Figure 1). No significant difference was observed for $\Delta L$ between the two ceramic types ($P=0.066$). On the other hand, the interaction effect of ceramic type and surface treatment on this parameter was not significant ($P=0.920$). However, surface treatment had a significant effect on $\Delta L$, and $\Delta L$ of polished specimens was higher than that of glazed specimens ($P=0.046$).

The mean and standard deviation of $a^*$ values in ceramic specimens before and after aging are presented in Table 2. The baseline $a^*$ in e-max Press specimens was significantly higher than that of Ceramco III specimens ($P<0.0001$); however, the difference between glazed and polished specimens was not significant ($P=0.105$). The interaction effect of ceramic type and surface treatment was not significant ($P=0.317$). After aging, the $a^*$ parameter was significantly higher in the e-max Press ceramics compared to the Ceramco III ($P<0.0001$). Type of surface treatment had no significant effect on the $a^*$ parameter ($P=0.326$). The interaction effect of ceramic type and surface treatment was not significant ($P=0.201$). The $\Delta a$ was positive for both ceramic types. The $\Delta a$ increased after aging and no significant difference detected for $\Delta a$ between the two ceramics ($P=0.670$). The two surface treatments showed no significant difference in this regard either ($P=0.558$). The interaction effect of ceramic type and surface treatment on this parameter was not significant ($P=0.456$).

The mean and standard deviations of $b^*$ values in ceramic specimens before and after aging is shown in Table 3. The baseline $b^*$ parameter in e-max Press specimens was significantly higher than Ceramco III ($P<0.0001$); however, no significant difference was observed between the glazed and polished specimens ($P=0.140$). The interaction effect of ceramic type and surface treatment on this parameter was not significant either ($P=0.226$). The two ceramic types were significantly different in $b^*$ parameter after aging ($P<0.0001$) and the $b^*$ value was higher for e-max Press compared to Ceramco III. Surface treatment had no significant effect on $b^*$ value ($P=0.164$); but the interaction effect of ceramic type and surface treatment on $b^*$ parameter was significant after aging ($P=0.03$).

The type of surface treatment had a significant effect on $b^*$ parameter for Ceramco III ceramics ($P=0.0001$). After aging, the $b^*$ parameter of glazed Ceramco III specimens was higher compared to polished Ceramco III specimens. This effect was not significant for e-max Press ceramics ($P=0.642$; Table 3). The $\Delta b$ was negative in all specimens. This means that after aging, the $b^*$ values decreased. The two ceramic types were not significantly different in terms of $\Delta b$ ($P=0.965$). Surface treatment had a significant effect on $\Delta b$ ($P=0.0001$). This parameter in polished specimens was higher than that of glazed specimens. The interaction effect of ceramic type and surface treatment on $\Delta b$ was also significant ($P=0.002$). The results of t-test showed that the difference in $\Delta b$ between glazed and polished specimens was greater in Ceramco III ceramic (compared to e.max Press). In other words, the highest and the lowest $\Delta b$ were found for the polished and glazed Ceramco III specimens, respectively. For the e-max Press ceramics, $\Delta b$ of polished specimens was higher than that of glazed specimens ($P=0.004$).

The mean and standard deviation of $\Delta E$ in the two ceramic types with the two surface treatments after 300 hours of aging are presented in Table 4. After aging, $\Delta E$ of the two ceramic types with two surface treatments ranged from 1.31 to 2.53. The interaction effect of ceramic type and surface treatment on $\Delta E$ was not significant ($P=0.828$). Although, a difference was noted in $\Delta E$ between the two types of ceramics, it was not statistically
**Table 1.** Mean and standard deviation of $L^*$ parameter of the two types of ceramics with two surface treatments before and after aging

<table>
<thead>
<tr>
<th>Material (n=10)</th>
<th>Surface treatment</th>
<th>$L^*$ baseline</th>
<th>$L^*$ aging</th>
<th>$\Delta L^*$</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPS emax-press</td>
<td>Polished</td>
<td>74.07±0.31</td>
<td>72.29±0.74</td>
<td>0.62±1.78</td>
</tr>
<tr>
<td></td>
<td>Glazed</td>
<td>74.07±1.20</td>
<td>73.24±1.12</td>
<td>0.40±1.23</td>
</tr>
<tr>
<td>Ceramco III</td>
<td>Polished</td>
<td>76.92±0.37</td>
<td>74.67±0.54</td>
<td>-2.25±0.28</td>
</tr>
<tr>
<td></td>
<td>Glazed</td>
<td>77.38±0.44</td>
<td>75.62±529</td>
<td>0.80±1.75</td>
</tr>
</tbody>
</table>

**Figure 1.** FE-SEM micrograph of glazed (above) and polished (below) IPS e-max Press. X500 magnification
significant (P=0.055). Also, ΔE of polished specimens was significantly higher than that of glazed specimens (P=0.018). Thus, in general, it may be stated that polished ceramics had a higher ΔE than glazed ceramics. Figures 1 and 2 show SEM micrographs of glazed and polished ceramic specimens.

As seen in Figure 2, polishing created a slightly rougher surface compared to glazing in IPS e-max Press ceramic, although this difference was not apparent for Ceramco III ceramic. Glazed surfaces were smooth; however, porosities were also noted.
Discussion
Considering the increasing emphasis placed on esthetics, color stability of esthetic dental restorative materials is critical. Ideal restorative materials must have optimal mechanical stability and excellent esthetic properties [7]. Two silica-based all ceramic systems were evaluated in the present study. Accelerated aging was performed to simulate the effects of long-term exposure of ceramics to environmental factors by UV light radiation and moisture and thermal alterations. The manufacturers of accelerated aging devices claim that 300 hours of aging corresponds to one year of clinical service [18]. Razzoog et al. [19] showed that the greatest color change occurs within the first 100 hours of accelerated aging. Although conditions are more complex in the oral cavity, such simulation of aging is useful for behavior comparison of different dental materials [19]. Most previous studies on color of tooth-colored restorative materials have used spectrophotometry and CIE L*a*b* color system introduced in 1976. The advantage of this system compared to other systems is that it enables interpretation of clinical results. Color change in this system is compatible with color perception by the human eye and by using this system, the clinically perceivable color change can be
determined. Thus, this system is accurate for color analysis and visual assessment errors rarely occur [20]. The efficacy and accuracy of spectrophotometry have been confirmed in previous reports [16, 21].

After 300 hours of aging, the L* parameter decreased in both ceramics and the specimens became darker. Also, aging increased the a* parameter or redness in both ceramics. The b* parameter also increased after aging and ceramic specimens showed a color shift towards blue. These changes in a* and b* parameters were observed in both ceramic types. These results were in agreement with those of Saygili and Demirel [20] regarding the same color change in restorative materials. Color of ceramics is influenced by the color change of metal oxides in their structure during the process of heating. These metal oxides are added to ceramic matrix to obtain a favorable color. Metal oxide bonds easily break down when exposed to UV light. By production of peroxidase compounds, color of ceramics significantly changes [22]. Studies have shown that some metal oxides undergo discoloration after heating in a furnace. Crispin et al. [6] and Lund and Pitrowski [23] reported that yellow and orange stains have minimal color stability at different baking temperatures. Also, Mulla and Wiener [22] showed that blue stain has minimal color stability in ceramic systems while orange stain has the highest color stability at different baking temperatures.

Less porosity in ceramic after laboratory phases results in greater color stability of ceramic. In other words, hand-made ceramic restorations have higher color stability than factory-made ceramics; although factors such as use of extrinsic dyes, presence of metal framework, number of heating cycles and baking in vacuum may affect the color stability of ceramics as well [24]. The ∆E of ceramic samples was in the range of 1.31 to 2.53 after 300 hours of aging in our study. Nakamora et al. [25] stated that the visually perceivable ∆E was 1.4, while Segui et al. [26] reported the clinically perceptible ∆E to be 2. The acceptable threshold for ∆E of crowns was considered to be 1.1 (in terms of difference in redness) by Douglas and Brewer [27]. Vichi et al. [28] defined the acceptable ∆E as ∆E values smaller than 1, which are not perceivable by the human eye. The ∆E values between 1 and 2 are detectable by non-skilled but meticulous observers and ∆E values >2 are perceivable by all non-skilled observers. Finishing and polishing techniques to obtain a smooth ceramic surface (comparable to the glazed ceramic) have been the topic of many previous studies. It is recommended to use polishing as an alternative to glazing. Some others compared polished and glazed surfaces using a profilometer, SEM and visual inspection. These studies reported that surface roughness of glazed and polished ceramic surfaces was comparable [11-15]. Haywood et al. [29] used a series of finishing tools with diamond grits followed by 30-fluted carbide burs and diamond polishing pastes for ceramic polishing and reported that this method yields a surface as smooth as a glazed surface. However, Patterson et al. [30] noticed that ceramic polishing tools left some voids in the ceramic surface while no such voids were detected in glazed surfaces. Wang et al. [31] stated that a smooth surface texture is also important for color of restorations since a smooth surface reflects greater amount of light compared to a rough surface and a rough or irregular texture scatters the light or causes irregular reflection of light and changes the color of restorations.

In our study, ∆L and ∆b of polished specimens were higher than those of glazed specimens. In other words, aging changed the L* and b* parameters in polished specimens more than glazed specimens. The stability of L* and b* parameters after accelerated aging was lower in polished specimens than in glazed specimens. Color change (∆E) of polished specimens was greater in comparison to the glazed specimens. This finding was in agreement with that of Kim et al [32]. They evaluated the effect of surface topography on color of dental ceramics and found that although surface roughness after polishing was not significantly different from surface roughness after glazing, the difference in color of glazed and polished specimens was significant. Color of dental ceramics is affected by factors other than surface roughness such as the bond between crystalline phases and glass matrix, coefficient of thermal expansion and presence of defect at the grain boundaries. Lee et al. [33] showed that surface topography affects the color of ceramics especially...
the L* parameter. Also, it has been reported that the a* and b* values increase after glazing of ceramics. Although glazed surfaces appear whiter, measurement of L* parameter by specular component excluded geometry shows lower values compared to polished surfaces. The difference between our results and those of other studies regarding the L* parameter may be due to the use of different polishing systems and ceramic materials. Furthermore, Sarac et al. [10] evaluated the effect of different polishing systems on the color and surface texture of feldspathic ceramics and reported that use of polishing kit alone yielded a surface as smooth as glazed surfaces. However, use of paste alone could not provide such a smooth surface. The color of polished and glazed surfaces was also significantly different but ∆E was <3.3 and therefore was within the clinically acceptable range. Fuzzi and Zaccheroni [34] evaluated the glazed and polished ceramics using SEM and profilometry and found that although surface roughness of polished ceramics was relatively low, a significant difference existed between surface smoothness and irradiance. Another study showed that the grit size of silicon carbide papers used for ceramic polishing affects the color change of ceramics [35]. In all-ceramics, polishing cannot provide a surface as smooth as glazed surfaces but size of crystals and size of grains play an important role in surface topography [36]. In this study, 400-1200 grit abrasive papers were used since previous studies have shown that these papers provide optimal smoothness in ceramic surfaces [11, 13, 15]. Orbegan and Goodlinkd [37] evaluated the effect of opaque layer and texture of ceramic on the color of metal ceramic restorations and found that smoothness or roughness of ceramic texture does not change the hue but glossiness of opaque layer changes the color from yellow to yellow-red. They showed that a dull opaque layer has a lower chroma, and a glossy ceramic surface has higher value than a rough surface. However, the authors did not explain why different surface textures change the lightness. Hamzah et al. [38] in 2016 evaluated the effect of accelerated aging on surface roughness and color stability of three indirect materials including translucent zirconia/resin LAVA nano ceramic and a group of two-layer discs with InCoris zirconia in the first layer and a hand-made feldspathic ceramic for the second (veneering) layer. After 300 hours of aging, they found no significant difference between groups in terms of color stability and ∆E.

**Conclusion**

In conclusion, findings of the current study showed that the *a* and *b* parameters in e.max Press specimens were higher at baseline before and after aging compared to that of Ceramco III specimens. However, the L* parameter in Ceramco III was higher than that of e.max Press. The ∆E in both ceramic types was in the range of 1.31 to 2.53, which is within the perceivable threshold for professionals. No significant difference was noted in color stability between the two ceramic types after 300 hrs of aging. The color stability (ΔE) of polished ceramic surfaces was lower than that of glazed surfaces; this was also true for Δb and ΔL. Color stability of silica-based ceramic systems was influenced by the surface texture and not the type of crystalline phase.

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