## RESEARCH ARTICLE

# Effect of thickness, cement shade, and coffee thermocycling on the optical properties of zirconia reinforced lithium silicate ceramic

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#### Abstract

**Objective:** To investigate the effect of thickness, cement shade, and coffee thermocycling (CTC) on the optical properties of zirconia reinforced lithium silicate (ZLS) paired with different shades of a resin cement.

**Materials and Methods:** Thirty ZLS specimens were prepared in two different thicknesses (0.8 and 1.5 mm) and three different resin cement shades (Tr, A2, and A3) were applied (n = 5). Color determinations were done before and after 5000 CTC by using a noncontact spectroradiometer. Color change due to CTC and relative translucency parameter (RTP) before and after CTC were calculated by using CIEDE2000. Data were analyzed using ANOVA and Bonferroni-corrected *t*-tests( $\alpha = 0.05$ ).

**Results:** Material thickness and resin cement shade (P < 0.001) affected baseline color. Material thickness affected color difference (P = 0.025). Thickness, resin cement shade, and CTC ( $P \le 0.0001$ ) affected RTP. The difference between the color changes of the 0.8- and 1.5-mm specimens combined with A2 shade cement after CTC was significant (P = 0.01). RTPs of all pairs decreased after CTC (P < 0.001).

**Conclusions:** Cement shade and material thickness affected the baseline color. The thickness of ZLS affected the color change after CTC only with A2 resin cement and the color change was less when the ZLS was thicker. CTC reduced the translucency of all pairs.

**Clinical Significance:** Clinicians and patients should be aware of a potential color change after long-term coffee consumption when zirconia reinforced lithium silicate is used particularly for laminate veneers with A2 shade of the tested resin cement.

#### KEYWORDS

CAD/CAM ceramic, coffee thermocycling, color change, resin cement, translucency, zirconia reinforced lithium silicate

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## 1 | INTRODUCTION

Computer aided design-computer aided manufacturing (CAD-CAM) technologies have revolutionized dentistry,<sup>1–3</sup> facilitating the use of materials in monolithic form.<sup>4–6</sup> Considering the high expectations of both patients and clinicians for esthetic restorations, new materials are developed and introduced continuously.<sup>7</sup> Among these materials, zirconia reinforced lithium silicate glass ceramic (Vita Suprinity; Ivoclar Vivadent, ZLS) has a unique structure that comprises 10 wt% of zirconium dissolved in a glassy matrix and a dual microstructure consisting of lithium metasilicate and lithium disilicate crystals.<sup>1,8</sup> ZLS combines the optical properties of glass ceramics with the mechanical properties of zirconia<sup>9</sup> and it can be used for the fabrication of partial or complete coverage restorations including laminate veneers and crowns.<sup>10</sup>

A natural appearance and an esthetically pleasing outcome cannot be achieved without considering the optical properties of materials,<sup>11</sup> which should match those of natural teeth.<sup>12-14</sup> The light reaching a tooth surface may be reflected, diffused, absorbed, or transmitted and for an acceptable shade match, a similar behavior is expected from the materials.<sup>3</sup> Translucency is a fundamental factor defining the esthetic outcomes of ceramics.<sup>14</sup> Higher translucency results in higher light transmission<sup>15</sup> and the thickness of the material significantly affects the translucency.<sup>14</sup> A translucent restoration's final color may also depend on the shade of the luting cement.<sup>16</sup> However, the effect of cement shade can be minimal depending on the thickness of the material.<sup>17,18</sup> Nevertheless, cement shade becomes critical when a dark underlying abutment that needs to be masked is present or while using a ceramic thinner than 1.5 mm.<sup>11,12,17,19</sup>

The use of spectroradiometers is common in dentistry,<sup>2,4,5,20,21</sup> and the color parameters are evaluated by using the CIELab system.<sup>22,23</sup> The CIEDE2000 color difference formula corrects the nonuniformity of CIELab system, while improving the correlation between visual judgments and instrumental values in general<sup>22,24</sup> and within dental tooth colors.<sup>25</sup>

Information on the optical properties of ZLS is limited<sup>2,4,5,7,9,13,26</sup> and how different cement shades affect the color of ZLS after cementation has not been studied. Compared with lithium disilicate, ZLS has a smaller crystal size that may lead to an improved translucency.<sup>26</sup> However, the translucency of ZLS was found similar to zirconia when fabricated at a crown thickness.<sup>2</sup> Therefore, the optical properties of ZLS should be further investigated. This in vitro study aimed to evaluate the effect of material thickness and resin cement shade on the color and translucency of ZLS, both before and after coffee thermocycling (CTC). The hypotheses were that (1) material thickness

and resin cement shade would affect the baseline color of ZLS joined to resin cement, and (2) material thickness, resin cement shade, and CTC would affect the color and translucency of ZLS joined to resin cement.

#### 2 | MATERIAL AND METHODS

A zirconia-reinforced lithium silicate glass–ceramic (Vita Suprinity, A2-HT, VITA Zahnfabrik, Bad Sackingen, Germany) (ZLS) (N = 30) combined with translucent (Tr), A2, and A3 resin cement shades were tested for optical properties before and after CTC (Table 1). Precrystallized ZLS blocks were sliced (Vari/cut VC-50, Leco Corporation, St Josephs, MI, USA) to obtain final rectangular plates 0.8 ( $\pm$  0.03) mm and 1.5 ( $\pm$ 0.03) mm in thickness. After cleaning the specimens ultrasonically in distilled water (15 min), crystallization firing was done (Programat EP5000, Ivoclar Vivadent AG, Liechtenstein, Austria) following the manufacturer's recommendations. Then, the specimens were polished for 20 s under running water (silicon carbide abrasive papers, #600) to remove surface irregularities. To check the final thickness, measurements were made at five different points using a digital micrometer (Digimatic Caliper, Mitutoyo MC, Kawasaki, Japan). Each ceramic thickness group was then divided into three subgroups (n = 5).

For the preparation of the ceramic surfaces, 5% hydrofluoric acid gel (IPS ceramic etching gel, Ivoclar Vivadent AG, Liechtenstein, Austria) was applied for 20 s to one side of each specimen. Surfaces were then treated with a silane-coupling agent (Monobond Plus, Ivoclar Vivadent AG, Liechtenstein, Austria) for 60 s. A stainless-steel metal ring (Stainless steel shim, Seastrom, Twin Falls, ID, USA) with a thickness of 0.2 mm was used to join the resin cement to zirconia reinforced lithium silicate glass ceramic specimens and to create 0.2 mm cement thickness. Vaseline was applied as a separating medium underneath the metal rings, and the metal rings were placed on each ceramic specimen,<sup>12</sup> and a self-adhesive resin cement (RelyX Unicem 2 Automix Self-Adhesive Resin Cement, 3 M ESPE GmbH, Neuss, Germany) was injected into the metal ring from its cartridge. To stabilize the specimens during polymerization, a mylar strip and a glass slab were placed over the specimens. The mylar strip and glass slab were held in correct position with finger pressure until light curing was completed. The resin cements were light cured with a LED light curing unit at 750 mW/cm<sup>2</sup> for 60 s (40 s through the glass slab and an additional 20 s through the ceramic). Luting was performed by the same operator (G.C.) at standard laboratory condition. Then the mylar strip and metal ring were removed gently from the ceramic

#### TABLE 1 List of materials used

Material	Classification	LOT number	Manufacturer
Vita Suprinity, A2-HT	Zirconia reinforced lithium silicate glass-ceramic	49,143	VITA Zahnfabrik, Bad Sackingen, Germany
RelyX Unicem 2 Automix, A2	Self-adhesive resin cement	627,036	3 M ESPE GmbH, Neuss, Germany
RelyX Unicem 2 Automix, A3	Self-adhesive resin cement	628,908	3 M ESPE GmbH, Neuss, Germany
RelyX Unicem 2 Automix, Tr	Self-adhesive resin cement	628,462	3 M ESPE GmbH, Neuss, Germany



surface and the specimens were evaluated once again for thickness. The specimens were stored in a dark container for 24 h at 37C° in high-humidity (95%) to complete polymerization before baseline spectral radiance measurements. These procedures were repeated for each shade of the resin cement.

Baseline spectral radiance measurements and reflectance spectra determinations of the ZLS specimens were performed on black  $(L^* = 9.1, a^* = -0.2, b^* = 0.3)$ , gray  $(L^* = 70.2, a^* = -1, b^* = 2)$ , and white  $(L^* = 93.7, a^* = -0.3, b^* = 2.9)$  backings at wavelengths between 380 nm to 780 nm at intervals of 2-nm. A noncontact spectroradiometer (SpectraScan PR705, Photo Research Inc., Chatsworth, CA, USA) positioned at 0 ° observation and 45° illumination (Thermo Oriel Models 66,904 and 69,911, Thero Oriel Instruments, CA, USA) was used for radiance measurements at a measuring aperture size of 1.1 mm.<sup>4,5</sup> The stability of radiance measurements was monitored by measuring a certified white reflectance standard (S3796A; Labsphere Inc. North Sutton, NH) before and after each specimen and averaged for reflectance calculations. Saturated sucrose solution was applied between each specimen and backing to obtain optical contact. Each specimen was measured on each backing four times and averaged. The reflectance calculations were then derived by a using previously reported equation.<sup>4,5</sup> Commission Internationale de l'Eclairage L\*, a\* and b\* color parameters were obtained for the CIE D65 Illuminant and the CIE Standard (2°) Human Observer.<sup>22</sup>

After baseline measurements, the specimens were exposed to 5000 thermocycles (5–55°C, 10 s transfer and 30 s dwell time) (Buchi 461 Water Bath, Buchi Corporation, New Castle, DE, USA) in coffee.<sup>4,5</sup> For the preparation of the coffee solution, 177 ml of water was mixed with 1 round tablespoon of coffee (Maxwell House, Kraft, Canada).<sup>4,5</sup> The coffee solutions in each bath were changed every 12 h. After CTC was completed, ceramic surfaces were gently brushed 10 times using a toothpaste and running water (Crest, Procter and Gamble, Cincinnati,

USA) to remove coffee extracts. The specimens were ultrasonically cleaned for 15 minutes and dried by using tissue paper. Then, similar to baseline measurements, radiance measurements and reflectance, color, and translucency calculations were repeated.

The color differences using the CIEDE2000 formula between specimens when on black and white backings were used for the relative translucency parameter (RTP) calculations of the specimens before and after  $\rm CTC.^{4,5}$ 

Baseline colors on grav were analyzed for dependence on ZLS thickness and cement shade using a 3-way repeated measures ANOVA, where color direction was the within-subject factor and thickness and cement shade were between-subjects factors. Upon finding a statistically significant dependence of these baseline color parameters on the specimen formation factors, the color differences between ZLS thickness values at each cement shade and between cement shades at each thickness will be determined for all possible combinations of involved specimens. The baseline color coordinates determined on a gray backing before CTC were used to calculate the color differences of all possible specimen combinations comparing the 2 ZLS thicknesses at each shade and comparing cement shades at each thickness. CIEDE2000 ( $\Delta E_{00}$ ) color difference formula was used for all color difference calculations.<sup>22,23</sup> The parametric factors (kL, kC, and kH) were each set at 1. These means were then compared to perceptibility and acceptability thresholds for dental tooth colors.<sup>26</sup> Overview of the test design is illustrated in Figure 1.

The color measurements were performed with a spectroradiometer and the validity of its measuring geometry for color measurements of ceramic specimens was previously reported.<sup>25</sup> The color changes and the RTP values were calculated using the CIEDE2000 formula.<sup>22,23,25</sup> For color change due to CTC, mean CIEDE2000 values and 95% confidence limits were calculated for each thickness and cement shade, and these means were compared to perceptibility and acceptability



**FIGURE 2** Means and 95% confidence intervals for initial color differences before CTC. Green line represents perceptibility threshold, whereas red line represents acceptability threshold.28



**FIGURE 3** Means and 95% confidence limits for color change caused by CTC for thickness and cement shade combinations. Green line represents the perceptibility threshold, whereas red line represents acceptability threshold.28

thresholds for dental tooth colors.<sup>27</sup> Further, a 2-way ANOVA was used to determine the dependence of the change in color on gray due to CTC for the thickness and cement shade main effects and their interaction.

Mean RTP values and 95% confidence limits were calculated for each thickness, cement shade, and CTC. A 3-way repeated measures ANOVA was used to analyze the RTP dataset, where the CTC was the within-subject factor and thickness and cement shade were the between-subjects factors. All ANOVAs followed the technique of determining an optimized response distribution and, for repeated measures, an optimized covariance structure by finding minimized Akaike and Bayesian information criteria.<sup>28</sup> Each ANOVA used the restricted maximum likelihood estimation method and, for repeated measures, the Satterthwaite degrees of freedom method (SAS Proprietary Software 9.3, SAS Institute Inc., Cary, NC, USA). For a main



**FIGURE 4** Mean RTP values and 95% confidence limits for material thickness-resin cement shade combinations. B, before coffee thermocycling; C, coffee thermocycled (CTC)

effect found to be uniquely statistically significant with more than 1 degree of freedom, Tukey tests were used to resolve pairwise comparisons ( $\alpha = 0.05$ ).

## 3 | RESULTS

For the baseline colors on gray, the optimized analysis method was found to involve a normal response distribution and a covariance structure of standard variance components. The Type III tests of fixed effects of the 3-way ANOVA of these baseline colors revealed no statistically significant 3-way interaction ( $F_{72}^4 = 0.94$ , P = 0.443) and statistically significance ( $P \le 0.022$ ) for each 2-way interaction. Therefore, the color differences between the colors at the 2 thickness values for each cement shade and between every pair of cement shade at each thickness were calculated (Figure 2), with perceptibility and acceptability thresholds.<sup>27</sup> For the color change due to CTC, the means and 95% confidence intervals are provided in Figure 3. For this dataset, the optimized analysis method was found to involve a normal response distribution and the 2-way ANOVA revealed that the interaction between material thickness and cement shade was not statistically significant ( $F_{24}^2$  = 2.97, P = 0.070), and that the main effect of cement shade was not significant ( $F_{24}^2 = 0.10$ , P = 0.905), but that the main effect of thickness was ( $F_{24}^1 = 5.74, P = 0.025$ ).

Figure 4 presents the means and 95% confidence intervals for RTP, with perceptibility and acceptability thresholds.<sup>27</sup> For RTP, the optimized analysis method was found to involve a lognormal response distribution and a covariance structure of standard variance components. Table 2 summarizes the results of 3-way ANOVA for RTP, where no significant interaction was found, yet each of the main effects was found to be highly significant. To resolve the significant main effect of the shade of the cement, the Tukey tests revealed significant differences between shade A3 and each of A2 and Tr (P < 0.0001), with no difference between shade A2 and Tr.

Source of variation	df of numerator	df of denominator	F	Р
Shade	2	48	61.04	<0.001
Thickness	1	48	696.52	<0.001
Condition	1	48	17.33	<0.001
Shade*Thickness	2	48	1.13	0.332
Shade*Condition	2	48	0.54	0.586
Thickness*Condition	1	48	3.55	0.066
Shade*Thickness*Condition	2	48	0.78	0.464

**TABLE 2**Summary of tests of fixedeffects for 3-way ANOVA of RTPdeterminations

## 4 | DISCUSSION

The first hypothesis was accepted because the baseline color of ZLScement combinations was affected from the material thicknesscement shade interaction. When the CIEDE2000 50% perceptibility (0.81) and acceptability (1.77) thresholds were considered,<sup>27</sup> the color difference between 0.8 and 1.5 mm-thick specimens for each resin cement shade was clinically acceptable, but perceptible. Therefore, the difference each tested cement shade makes in color between ZLS in tested thicknesses may not be clinically significant. However, at each thickness, using A3 shade resin cement resulted in a clinically different (color difference above acceptability) ZLS color compared with when A2 or Tr-shade resin cements were used. The difference between A2 and Tr was acceptable, but perceptible, regardless of the thickness (Figure 2). Clinicians may consider using lighter shades (A2 and Tr) for a ZLS restoration at tested thicknesses as lighter shades' effects on ZLS' color were small. However, using the tested darker shade (A3) may allow masking a dark underlying structure, which should be investigated in the future.

In a previous study evaluating the color of lithium disilicate paired with cements in different brands and shades, both parameters were found effective on the color change.<sup>11</sup> Dede et al.<sup>12</sup> also reported that the final color of lithium disilicate was affected by the resin cement shade and the color of the underlying structure. Gürdal et al.<sup>7</sup> studied the effects of resin cement on the color of CAD-CAM materials in different thicknesses (0.5 and 1 mm), including ZLS, after thermocycling and reported that the material type and cement had an effect on the color change. However, the authors<sup>7</sup> did not specify the shade of the resin cements used or the baseline color measurements. Thus, to authors' knowledge, the present study is the first on the baseline color differences of ZLS paired with different shades of a resin cement. However, the present study did not investigate the color difference of ZLS with and without resin cement, and future studies involving this parameter can further elaborate the optical properties and masking ability of ZLS.

The second hypothesis was accepted as the thickness had an effect on color when paired with A2 shade resin cement and the effects of the thickness, shade, and CTC on RTP were found significant. The RTP values of 0.8 mm-thick specimens was higher than that of 1.5 mm specimens. In general, lighter cement shade resulted in higher translucency, but, the difference between Tr and A2 shades for 1.5 mm-thick ZLS was not significant. The CTC decreased the RTP of all cement shade-material thickness combinations.

Previous studies have reported that the color of CAD-CAM materials is affected by the resin cement shade, brand, and type.<sup>7,11,12,19</sup> Color change of ZLS paired with different shades of resin cement has not been extensively studied and only two studies were found.<sup>7,13</sup> Kandil et al.<sup>13</sup> studied the color difference of 0.5 mm-thick ZLS and hybrid ceramic in two different translucencies. The specimens were cemented on a dark background (C3) using A1 or opaque shaded resin cements. Resin cement shade had a significant effect and the opaque resin cement combined with the low-translucency type of both restorative materials resulted in a decrease in the color difference from the target shade (A1). The fact that thinner (0.5 mm vs. 0.8 mm in the present study) specimens were used in Kandil et al's.<sup>13</sup> study may be the reason for the difference in findings of their study and the present study. The difference in methodology may also be the reason for contradicting findings as Kandil et al's. study did not involve aging.<sup>13</sup> Color change in ZLS with different types of resin cements was also investigated and the effect of resin cement type was shown to be significant.<sup>7</sup> However, the cement shade's effect was not investigated. ZLS was in 0.5 and 1.0 mm thicknesses and the thermocycling duration in distilled water was 5000 cycles. The color change values after thermocycling were higher compared to those found in the present study, which may be due to the differences in cements and thicknesses tested.

After CTC, all color changes were above the perceptibility threshold except for when 1.5 mm ZLS was paired with A2 shade cement, and all mean color changes were below the clinically acceptable threshold (Figure 3). Arif et al.<sup>2</sup> analyzed color changes with ZLS and lithium disilicate after CTC using two different thicknesses (0.7 and 1.5 mm) and concluded that no difference in color change was observed between 2 thicknesses of the same material or between 2 materials for the crown thickness. In addition, the color change with ZLS was below the acceptability threshold and specimens prepared in complete crown thickness (1.5 mm) showed a color change lower than the perceptibility threshold. The differences in results for clinical acceptability and perceptibility may be due to different thresholds used and because Arif et al's.<sup>2</sup> study did not involve a resin cement. In another study, the color of 1.5 mm-thick ZLS and lithium disilicate ceramic after different surface finishing protocols was evaluated.<sup>4</sup> It was concluded that the effect polishing or glazing on the color change of ZLS after CTC was not significant and the color changes were not perceivable.<sup>4</sup> Again, no cementation was performed, which may be the reason for differences from the present study results in terms of perceptibility. Ozen et al.<sup>29</sup> studied the color stability of CAD-CAM

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materials after cementation with a translucent shade resin cement and thermocycling. The effect of translucent cement applied on the color of 0.5 mm ZLS specimens was similar to the effect of the cements recorded after cementation in the present study. However, a higher color change between after cementation and thermocycling was reported compared with the color change observed after thermocycling in the present study. The difference in findings regarding the color change due to thermocycling may be because of the fact that the specimens in the previous study were thinner than the specimens in the present study. In addition, a colorimeter was used in the previous study which is more prone to edge loss effect compared with the spectroradiometer used in the present study.

A previous study showed that CTC decreased the RTP of ZLS and lithium disilicate ceramic significantly at both glazed and polished states.<sup>4</sup> Moreover, it was also reported that polishing or glazing did not affect the translucency of ZLS.<sup>4</sup> The translucency of these materials was further elaborated by Arif et al.,<sup>2</sup> where the translucency of ZLS in laminate veneer and complete crown thicknesses was affected by CTC. However, this result contradicts with the findings of an earlier study reporting the nonsignificant effect of CTC on RTP values.<sup>5</sup> Nevertheless, both studies revealed higher translucency for thinner ZLS specimens. Comparing these findings to those in the present study may be problematic, since these studies did not involve resin cements. The translucency of ZLS might be the reason for the minor effect of the cement shade on the color change, as it has shown to be lower than that of lithium disilicate and similar to that of zirconia.<sup>2</sup> The low translucency of the material may mask the changes that may be seen in the cement's color due to CTC. The reason for the lower translucency may be due to the zirconia reinforcement in the structure of ZLS, which should be further studied.

In the present study, both surfaces of the specimens were exposed to staining through CTC. However, only one surface of a restoration is in contact with staining solutions clinically and the intaglio surface is cemented to the tooth surface.<sup>5,12</sup> Therefore, the findings of the present study should be carefully interpreted, and it may be speculated that the changes in optical properties after thermocycling/ clinical use would potentially be less when only one surface is exposed to coffee. Future clinical studies should be performed to investigate the long-term performance of tested materials. In addition, only one type of resin cement, which is a self-adhesive dual cure resin cement was used. Future studies are necessary to investigate the effects of underlying structures and resin cements of different curing and adhesion methods, to thoroughly understand the optical behavior of ZLS.

#### 5 | CONCLUSIONS

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

1. Material thickness and resin cement shade had a significant effect on the baseline color of ZLS joined to resin cement.

2. After CTC, the thickness of the ZLS affected the color change only when paired with A2 shade cement, and the thicker ZLS had improved color stability. 3. After CTC, lighter resin cement shade and thinner material resulted in higher translucency. However, the translucency of 1.5 mm thick ZLS was similar when combined with Tr or A2 shade resin cements.

4. CTC significantly decreased the translucency of all thicknessresin cement shade pairs.

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#### DATA AVAILABILITY STATEMENT

The data sets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

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