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RESEARCH AND EDUCATION

Effect of coloring liquids on color of zirconia frameworks and bond strength of zirconia/veneering ceramic

Selin Celik, DDS,[a] Yurdanur Ucar, DDS, PhD,[b] and Orhun Ekren, DDS, PhD[c]

ABSTRACT

Statement of problem. Zirconia substructures must be colored to fabricate esthetic restorations. However, the coloring procedure may affect the color match and bond strength of the veneering ceramic to the zirconia.

Purpose. The purpose of this in vitro study was to evaluate the bond strength of veneering ceramic to zirconia after dipping in coloring liquid for different durations and to compare the color of dipped blocks with precolored blocks.

Material and methods. Sixty-four cube-shaped specimens were fabricated from uncolored zirconia blocks by using computer-aided design and computer-aided manufacturing and divided into 4 groups (n=16). The G30 group was subjected to coloring liquids for 30 seconds, G60 for 60 seconds, and G120 for 120 seconds. After the coloring procedures, the color of the specimens was evaluated by using a spectrophotometer. Precolored zirconia (PCZ) was used as the control. Once the color measurements were completed, the same specimens were prepared for the bond strength test, with 2 groups as the control: PCZ and the uncolored control group (control bond-strength [CBS]). Veneering ceramic was applied to the zirconia specimens and sintered. The shear bond test was conducted using universal testing equipment with a crosshead speed of 1 mm/min. Statistical analysis was carried out by using 1-way ANOVA followed by the Tukey HSD test (α=.05). A Weibull analysis was performed, and the Weibull modulus was calculated for each group.

Results. The mean E value for the PCZ group (77.34 ±0.22) was statistically significantly different from that of all groups (PCZ-G30; P=.018, PCZ-G60; P= <.001, PCZ-G120; P<.001). The mean E value for G60 group was significantly different from mean E values of the G30 and G120 groups (G60-G30; P<.001, G60-G120; P<.001). The PCZ group had the highest mean shear bond strength value (31.5 ±7.67 MPa) and was significantly different from the mean bond strength values of the G120 and CBS groups (PCZ-G120; P=.008, PCZ-CBS; P<.001). The CBS had the lowest mean bond strength value (16.62 ±7.11 MPa) and was statistically significantly lower than that of the G60 group (CBS-G60; P=.001). The Weibull modulus was 64.78 for the PCZ group and 0.1 for the G120 group.

Conclusions. Immersing zirconia in coloring liquid altered the color and negatively affected the bond strength between the veneering ceramic and zirconia. (J Prosthet Dent 2020;124:110-5)

Ceramic restorations have become popular with the claim that better esthetic results are achieved than with metal-ceramic restorations.[1] Advances have included reinforced dental ceramics, including zirconia and lithium disilicate with improved mechanical properties.[2-5] Zirconia, fabricated with computer-aided design and computer-aided manufacturing, has excellent mechanical properties and is a material of choice especially in high stress-bearing areas.[6-9]

Zirconia blocks are chalk white with limited translucency.[10] To improve color match, zirconia cores can be colored and veneered with feldspathic ceramic.[10] However, zirconia veneers are liable to fracture,[10-16] leading to the popularity of anatomic contour zirconia, even though these restorations are less esthetic.[17] Zirconia can be colored with 2 processes[18]: colored pigments can be mixed with zirconia particles and pressed to form precolored zirconia (PCZ) blocks, setting the color during manufacturing[19]; alternatively the milled zirconia

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[a]Research Assistant, Cukurova University Faculty Of Dentistry, Department of Prosthodontics, Adana, Turkey.
[b]Professor, Cukurova University Faculty Of Dentistry, Department of Prosthodontics, Adana, Turkey.
[c]Associate Professor, Cukurova University Faculty Of Dentistry, Department of Prosthodontics, Adana, Turkey.

Clinical Implications

Using coloring liquid to shade zirconia, restorations may result in a less predictable color match and decrease the bond strength between zirconia and veneering ceramic.

can be dipped in coloring liquids containing metal oxides before sintering.[20,21] However, the immersion type coloring process is difficult to control, with excess ions penetrating the green zirconia and producing an unwanted shade. Excess metal ions on the surface of zirconia act as impurities and may adversely affect the bond with the veneering ceramic.[19] However, studies on how the coloring liquid affects the color match and the bond strength of veneering ceramics to zirconia are sparse. Kim and Kim[20] reported that the number of coloring liquid applications affects the lightness, yellow chromaticity, and opalescence of monolithic zirconia, although its translucency was unaffected by the coloring procedure. Ekren[22] compared the color of precolored and liquid-colored zirconia and reported a color match if the manufacturer's recommendations were followed. Atash et al[23] examined the effect of coloring liquid on the adhesion of veneering ceramic to zirconia. They used a 3-point flexural strength test and concluded that the bond strength between veneering ceramic and zirconia was affected by coloring liquid. However, Aktas et al[24] reported that coloring zirconia did not affect the adhesion of veneering ceramic to zirconia, but quality of veneering ceramic is crucial on adhesion to the zirconia substructure.

Macrotensile and microtensile tests have been used to measure bond strength. The microtensile test has been preferred for offering a more uniform stress distribution at the bonded surface.[25] Despite advantages, preparing microtensile test specimens is a technique sensitive process, as an interfacial crack can easily be created, which initiates crack propagation and results in premature failure at a lower strength.[25] Preparation of test specimens for the macroshear bond strength (macro-SBS) test is straightforward and rapid, with no additional specimen preparation after the bonding procedure. However, macro-SBS has disadvantages, including uneven stress distribution across the interface.[25]

The purpose this in vitro study was to evaluate the bond strength of veneering ceramic to zirconia after dipping in coloring liquid at different times and to compare the color (E) of dipped blocks with that of PCZ. The null hypotheses were that no difference in terms of color would be found between the PCZ and blocks colored with liquid and that the coloring liquid would have no effect on the bond strength between veneering ceramic and zirconia.

MATERIAL AND METHODS

Power analysis was performed using the G Power 3.0.1 software program. For color evaluation effect, the size (f) was 0.5, power was 0.85, and 4 groups were compared. The total number of specimens was determined to be 54. A total of 64 specimens (n=16) were used for color analysis.

For mechanical test effect, the size (f) was 0.5, power was 0.85, and 5 groups were compared. As per these parameters, the total sample size was 60 (n=12) for mechanical tests. Data from 12 specimens were included in the statistical analysis for the control bond-strength (CBS) group. The sample size was n=13 for all other groups.

An 8×8×8-mm cube was designed digitally (CEREC inLab SW 16.0; Dentsply Sirona), and 64 zirconia specimens were fabricated from uncolored zirconia blocks (Rainbow Zirconia Block; Genoss) with a computer-aided manufacturing unit (CEREC inLab MCX5; Dentsply Sirona) and divided into 4 groups (n=16/group). The cubes were colored as per the manufacturer's protocol. Specimens in the G30 group were subjected to coloring liquid (Rainbow Coloring Liquid DP2; Dentium) for 30 seconds, specimens in the G60 group for 60 seconds, and specimens in the G120 group for 120 seconds (the manufacturer's recommendation for the desired shade). After being dipped in coloring liquid, the specimens were sintered for10 hours at 1150 °C (inFire HTC Speed; Dentsply Sirona) as per the manufacturer's recommendations. CBS was the control group for the macro-SBS test and was not colored. In addition, cube-shaped PCZ (Rainbow Shade Block A2; Genoss) specimens were fabricated (n=16) as the control group for color measurement and the macro-SBS test. The color of the test specimens was evaluated using a spectrophotometer (CM-3600A; Konica Minolta). Commission Internationale de l'Eclairage 1976 L*a*b* coordinates were recorded on 5 different measurement points for each specimen. The diameter of each measurement was 4 mm. The color coordinates of each specimen were calculated by the mean L* a* b* values of 5 measurements per specimen; 4 points were placed on the corners and 1 on the center. The color of each group was determined by calculating the mean E value of 16 specimens. E value was calculated using the equation $E = \sqrt{L^{*2}+a^{*2}+b^{*2}}$. One test specimen from each group was selected and subjected to scanning electron microscopy-energy dispersive spectroscopy analyses before ceramic application.

Once the color evaluation was completed, specimens (n=16) were prepared for veneering ceramic application. Liner (Rainbow Liner A2; Genoss) was applied on the center of each specimen surface. Veneering ceramic (Rainbow Dentin Porcelain A2;

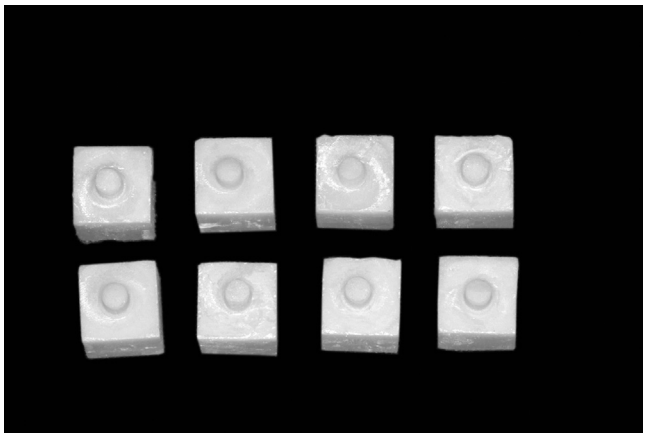


Figure 1. Veneering ceramic fired on zirconia substructures.

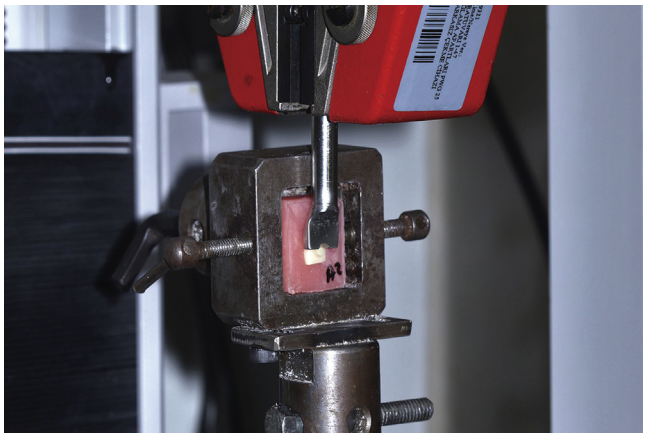


Figure 2. Macroshear bond strength test assembly.

Table 1. E Values of Test Groups

Group	E Value ±SD	Groups	P
PCZ	77.34 ±0.22	G30	.018
		G60	<.001
		G120	<.001
G30	78.16 ±0.95	PCZ	.018
		G60	<.001
		G120	.515
G60	81.16 ±0.77	PCZ	<.001
		G30	<.001
		G120	<.001
G120	78.54 ±0.88	PCZ	<.001
		G30	.515
		G60	<.001

PCZ, precolored zirconia SD, standard deviation.

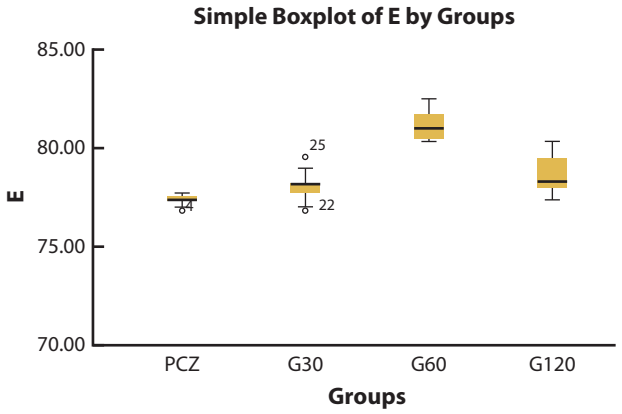


Figure 3. Color values.

Genoss) with a 3-mm diameter and 3-mm thickness was applied on the center of each zirconia and sintered (Fig. 1), and the specimens were embedded in acrylic resin (Meliodent RR; Kulzer GmbH). The macro-SBS test was performed using universal testing equipment (M500-25KN; Testometric) at a crosshead speed of 1 mm/min until fracture occurred (Fig. 2). Outlier values were omitted, resulting in 13 specimens for each group except CBS (n=12).

Data analyses of color (E) and bond strength values were evaluated by using 1-way ANOVA followed by the Tukey HSD test (α=.05). Weibull analysis was conducted, and Weibull modulus was calculated. A statistical software program (IBM SPSS Statistics, v21.0; IBM Corp) was used for the data analysis.

RESULTS

Means and standard deviations of the E values of tested groups as per 1-way ANOVA (dF:4, F:7.992, *P*<.001) are given in Table 1 and Figure 3. The color of PCZ was statistically significantly different than in all other test groups. Although there was a statistically significant color difference between the G30 and G60 groups (*P*<.001), no statistically significant difference was observed between the G30 and G120 groups (*P*=.515).

The mean bond strength values between veneering ceramic and zirconia are given in Table 2 and Figure 4. The highest mean bond strength value was found for PCZ (31.5 ±7.67 MPa) and was significantly higher than for the CBS and G120 groups. The lowest mean bond strength value was seen in the CBS group (16.62 ±7.11 MPa) and was statistically significantly different than G60 and PCZ.

Typically, fracture data are not normally distributed because of the brittleness of the materials. In accordance with the Weibull analysis, the highest Weibull modulus was observed for PCZ (m=64.78), and data were distributed uniformly. Prolonged dipping times in coloring liquid caused nonhomogeneous distribution as observed for the G120 group, where the lowest Weibull modulus was seen (m=0.1) (Fig. 5).

As seen in the representative energy dispersive spectroscopy spectra, zirconia specimens colored with coloring liquid showed significant peaks for aluminum (Al) and silicon (Si), yielding significant evidence of the

Table 2. Bond Strength Values of Groups

Group	Bond Strength ±SD (MPa)	Group	*P*
CBS	16.62 ±7.11	G30	.086
		G60	.001
		G120	.471
		PCZ	<.001
G30	24.26 ±6.98	CBS	.086
		G60	.535
		G120	.874
		PCZ	.101
G60	28.75 ±7.80	CBS	.001
		G30	.535
		G120	.103
		PCZ	.870
G120	21.50 ±7.22	CBS	.471
		G30	.874
		G60	.103
		PCZ	.008
PCZ	31.50 ±7.67	CBS	<.001
		G30	.101
		G60	.870
		G120	.008

PCZ, precolored zirconia; SD, standard deviation.

presence of these elements besides the elements in yttria-stabilized tetragonal zirconia polycrystal (Fig. 6).

DISCUSSION

Based on the findings of this in vitro study evaluating the effects of coloring procedures (liquid-shaded or precolored) on the color of zirconia frameworks and the bond strength between zirconia and veneering ceramic, the null hypotheses were not accepted.

The coloring liquid used in the present study was designed to reproduce the Vita A2 shade, with immersion of green zirconia for 2 minutes (G120) as per the manufacturer's recommendation for matching the Vita A2 shade. However, the Vita A2 specimens in the PCZ group were significantly different from the color of the liquid-shaded zirconia groups (G30, G60, and G120). Ekren[22] used a similar methodology for comparing color differences as in the present study, with the same brand of zirconia blocks and coloring liquids but with different immersion periods (120, 240, and 360 seconds). He reported no significant color difference between PCZ blocks and liquid-shaded zirconia blocks if manufacturer recommendations were followed. However, the results of the present study were not consistent with those of Ekren's[22] study, and, although the manufacturer's recommendations were followed, the coloring procedures might produce unpredictable color matches. Kim and Kim,[20] instead of dipping, brushed the coloring liquid on the uncolored zirconia 1 time, 2 times, and 3 times to improve control over the procedure. They concluded that increased application of coloring liquid reduced the

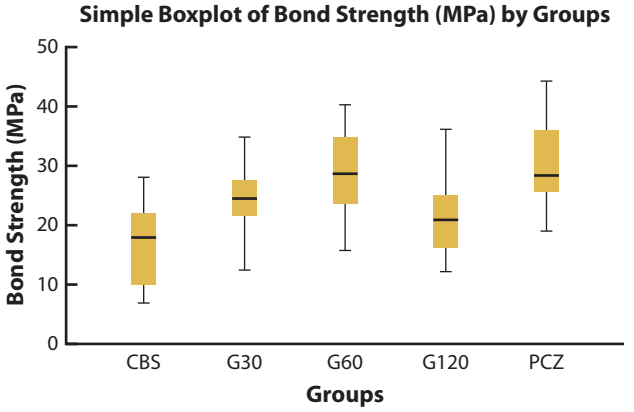


Figure 4. Bond strength values.

lightness and opalescence and increased yellowness. However, Kim et al[7] reported that the type of coloring agent and the number of applications did not affect the color of zirconia.

The results of the present study are consistent with those of the study by Aboushelib et al,[19] who reported 31.6 ±7.7 MPa and 30.1 ±6.9 MPa bond strength for 2 different PCZs. They also concluded that coloring pigments concentrated on the outer surface may be responsible for weakening the bond between the zirconia and the veneering ceramic.[19] In the present study, aluminum (Al) and silicon (Si) ions were detected on the surface of the zirconia, which supports the findings of Aboushelib et al.[19] Atash et al[23] recently investigated the bond strength of veneering ceramics to zirconia substructures colored with coloring liquid by using the 3-point flexural strength test and reported a 46 ±5.03 MPa bond strength value to uncolored zirconia and 52.41 ±8.87 MPa to liquid-colored zirconia. They concluded that coloring liquids can significantly affect the bond strength of veneering ceramic to zirconia. Aktas et al[24] reported bond strength values of different veneering ceramics to various zirconia substructures of between 27.1 ±4.1 MPa and 39.7 ±4.7 MPa and concluded that coloring zirconia did not significantly impair bonding of veneering ceramic.

Uneven distribution of stress across the interface is inevitable during macro-SBS testing.[25] To address this problem, a crescent-shaped rod was used in the present study instead of a knife-edged rod. A crescent-shaped rod can better dissipate the stress to a larger area, thus reducing uneven stress concentrations.[25]

Studies evaluating the effect of coloring liquids are limited,[22-24] and a Weibull analysis on the strength data is lacking. Therefore, a comparison of the Weibull data with previous studies was not possible. The prefabricated shaded group had the highest Weibull modulus (m=64.78), indicating less variability among the specimens. This group also had the highest Lnσ value. The R^2 value of this group was the highest, yielding much better

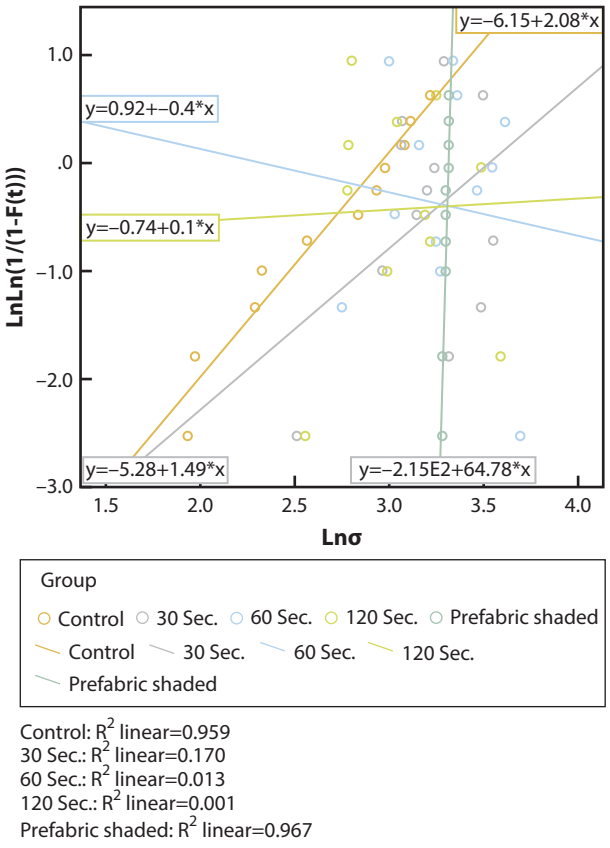


Figure 5. Weibull plot of bond strength test specimens.

fitting data on the line. Unlike the prefabricated group, specimens dipped in coloring liquid had lower Weibull modulus values and spread Lnσ data, indicating increased deviation. The R^2 values of the dipped specimens were lower. These results indicate the unpredictability of the mechanical properties of dipped specimens. Specimens in the control groups had more improved properties than the dipped specimens. However, the properties of CBS were not as good as in the PCZ group.

A standard method of specimen preparation for color measurement of zirconia ceramics is lacking. Different specimen dimensions have been reported, either a cube or disk shape.[7-9] To stabilize the specimens during the macro-SBS test, 8×8×8-mm cube shaped zirconia was fabricated for the present study.

Limitations of the present study included that no aging process was used on the test specimens before the shear test and that the ceramic was fired as in actual laboratory practice. However, repeated ceramic firings for dentin, enamel, and glazing to create esthetic restorations and aging may further deteriorate the bond strength. In addition, the chemical compositions of the coloring liquids were not analyzed. Comparison of the effect of different coloring liquids and analyzing the chemical composition of the coloring liquid are planned for future studies.

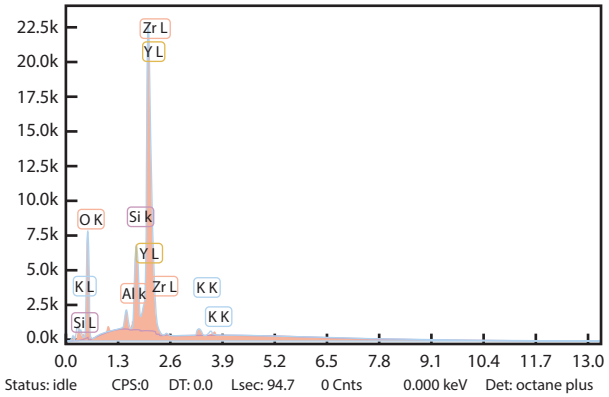


Figure 6. Energy dispersive spectroscopy spectra of zirconia substructure colored with coloring liquid.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Immersion of green zirconia in coloring liquids is not a predictable process for obtaining the desired shade.
2. Immersion in coloring liquid also negatively affects the bond strength of veneering ceramic to the zirconia substructure.
3. Further studies are needed to understand the effects of the coloring process on the physical properties of zirconia.

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Corresponding author:
Dr Orhun Ekren
Cukurova University
Faculty of Dentistry
Department of Prosthodontics
Balcalı/Sarıcam, Adana
TURKEY
Email: oekren@cu.edu.tr

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