

RESEARCH AND EDUCATION

Effects of treatment with aqueous and acid-based coloring liquid on the color of zirconia



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Color reproduction with monolithic zirconia restorations is challenging because of the opaque nature of this material. As zirconia is primarily white in color, it exhibits a higher brightness and lower saturation than natural tooth color and must be colored to simulate the natural tooth structure.¹⁻³ Zirconia can be colored with additive or painting techniques. For the adding techniques, a metal oxide colorant is mixed with the zirconia powder during manufacture of the zirconia block, providing a defined color from the initial stages.⁴⁻⁶ In contrast, the painting technique involves the presintering of zirconia powder to produce a block, followed by the application of various coloring agents to obtain the desired color.⁴⁻⁶

Although these processes provide colored zirconia, they do not allow for the color to be matched to the individual characteristics of the patient. In addition, metal oxides affect the physical properties of zirconia, including a strength reduction of the material.⁷ Recently, layered zirconia blocks consisting of a stratified structure of dentin color and transparent enamel have been developed.⁸ These blocks also use an additional coloring agent to match the characteristics of each patient in clinical practice.

In terms of the agents used in zirconia coloring, both acid-based coloring liquids and aqueous coloring liquids

ABSTRACT

Statement of problem. To preserve the mechanical property of color-treated zirconia for optimal restoration longevity, aqueous colorants have been developed as an alternative to acid-based coloring agents. However, little is known regarding the effects of aqueous colorants on the color of zirconia.

Purpose. The purpose of this in vitro study was to compare the effect of aqueous coloring liquids with acid-based coloring liquids on the color of zirconia.

Material and methods. Eighty monolithic zirconia specimens (10×10×2 mm) were fabricated and divided into 4 groups according to their color treatment: unshaded zirconia (control), precolored zirconia, aqueous coloring liquid on zirconia, and acid-based coloring liquid on zirconia. The shaded zirconia specimens were further divided into 3 subgroups based on the number of coloring applications used (1, 3, or 6). The International Commission on Illumination (CIE Lab) color coordinates were measured by spectrophotometry.

Results. Significant differences in the CIE a^* and b^* values were observed between the specimen groups treated with the aqueous coloring liquid and the acid-based coloring liquid ($P<.001$). Increasing the number of colorings resulted in an increase in the CIE a^* and b^* values and a decrease in the CIE L^* values in all the groups ($P<.001$).

Conclusions. Treatment with aqueous coloring liquid on zirconia produced a greater redness or yellowness compared with treatment with acid-based coloring liquid. The coloring of zirconia lowered its brightness and imparted a red/yellow hue. (J Prosthet Dent 2019;121:363.e1-e5)

have been commonly used. The acid-based coloring agent is composed of a solution of a strong acid such as hydrogen chloride (HCl) at pH 1 to 3 to which the desired metal oxides are added. The resulting acid-base reaction allows the color of zirconia to be adjusted. However, acid-based coloring agents are undesirable, as they can release acid vapors.⁹ Furthermore, the mechanical strength of colored zirconia appears to be reduced when such colorants are applied.¹⁰

Aqueous colorants have been developed that exclude the acid component, instead allowing the metal cations to permeate the porous zirconia and impart the desired color.^{9,11} The hardness of zirconia treated with aqueous

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Clinical Implications

Three applications of aqueous coloring liquid and acid-based coloring liquid was most similar to the natural tooth color but had increased yellowness, and the aqueous coloring liquid exhibited greater yellowness. This should be considered in clinical practice.

coloring liquid is significantly higher than that of specimens treated with acid-based coloring liquid.¹² Although there is a growing interest in aqueous colorants, little is known about their effects on the color of zirconia.

Seok et al¹³ analyzed the elution components of colored zirconia. In the case of the Zirkozahn aqueous coloring liquid, the Fe component was determined to be 0.03201 ppm, which was higher than those of the acid-based or aqueous coloring liquids provided by other manufacturers. In addition, Kaya¹⁴ found that higher Fe₂O₃ contents gave higher CIE b* values.

According to Kim et al,¹⁵ the CIE L* value decreased and the CIE b* value increased as the number of coloring applications increased for both aqueous and acidic coloring agents. The aqueous coloring liquid resulted in a greater yellowness (CIE b*) as the number of coloring applications increased. Kim and Kim¹⁶ reported that the CIE L* and opalescence parameter values decreased and the CIE b* value increased upon increasing the number of hydrocarbon-based coloring liquid applications.

The purpose of the present study was to investigate the effect of aqueous colorants on the color of zirconia. The null hypotheses were that the type of coloring liquid used does not affect the color of zirconia, that the number of coloring liquid applications does not affect the color of zirconia, and that there is no interaction effect between the type of coloring liquid and the number of coloring liquid applications.

MATERIAL AND METHODS

The specimens were designed using SolidWorks design software (Dassault Systèmes SolidWorks). The presintered zirconia block was cut using a Zirkozahn M5 milling unit (Zirkozahn GmbH). Taking into consideration a shrinkage of 20%, a total of 80 specimens measuring 13×3×2.3 mm were fabricated and separated into groups of 10 specimens (Table 1). The roughness of each specimen was adjusted using 1000-grit silicon carbide paper. The experimental group consisted of precolored zirconia specimens (Alpha Z 3; DMAX) and zirconia specimens (Alpha Z 0; DMAX) painted with the acid-based (Color Liquid Prettau A3; Zirkozahn GmbH) and the aqueous (Color Liquid Prettau Aqua-rell A3; Zirkozahn GmbH) coloring liquids, which

Table 1. Specimen groups according to color treatment (n=10)

Material	Coloring Liquid	Number of Coloring Liquid Applications	Group Code
Alpha Z 0	-	-	Z
Alpha Z 3	-	-	CZ
Alpha Z 0	Acid based	1	ZA1
		3	ZA3
		6	ZA6
Alpha Z 0	Aqueous	1	ZW1
		3	ZW3
		6	ZW6

CZ, precolored zirconia; Z, unshaded zirconia (control); ZA1, acid-based coloring liquid, 1 application; ZA3, acid-based coloring liquid, 3 applications; ZA6, acid-based coloring liquid, 6 applications; ZW1, aqueous coloring liquid, 1 application; ZW3, aqueous coloring liquid, 3 applications; ZW6, aqueous coloring liquid, 6 applications.

were applied to the specimen 1, 3, or 6 times with a synthetic nylon fiber brush (Hwahong 948; Hwasung-si). An unshaded zirconia block was used for the control group.

The specimens were dried for 30 minutes in an oven at 130°C before sintering in a furnace (LHT 02/17LB; Nabertherm) according to the manufacturer's instructions. The temperature of the furnace was raised to 1000°C for 3 hours and maintained for 1 hour and then raised to 1550°C for 3 hours and maintained for 2 hours. Finally, the specimens were ground using 1000-grit silicon carbide paper to give a final specimen size of 10×10×2 mm.

To observe the color differences among the various zirconia specimens, unshaded zirconia specimens (Z) were coated with acid-based (ZA) and aqueous (ZW) coloring liquids using 1, 3, or 6 coatings, and the resulting colors were measured. The colors of the final sintered zirconia specimens were measured using a cordless hand-held spectrophotometer (colorimeter) with a 5-mm-diameter contact probe tip (VITA Easyshade Advance 4.0; VITA Zahnfabrik) under identical conditions. This system captured the color coordinates using a D65 illuminant (color temperature 6500 Kelvin; a mathematical construct equivalent to average daylight in the northern hemisphere). The display showed the closest VITA shade in the VITA classical A1 to D4 or VITA 3DMaster shade guide.

The specimen was placed on a white plate, the CIE L*, a*, and b* values were measured, and the ΔE* value was calculated according to the following equation:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

$$(\Delta L^* = L_1^* - L_2^*, \Delta a^* = a_1^* - a_2^*, \Delta b^* = b_1^* - b_2^*).$$

The value of L* is expressed as 0 (black) to 100 (white) and represents brightness. a* indicates the degree of red and green, (+) indicates red, (0) indicates gray, and (-) indicates green. b* indicates the degree of yellow and

Table 2. CIE L*, a*, b* values of zirconia specimens depending on application of coloring liquid (±SD)

Group	L*	a*	b*
Z	94.1 ±0.2 ^a	-0.9 ±0.1 ^f	5.9 ±0.2 ^g
CZ	85.5 ±0.8 ^b	2.9 ±0.9 ^d	33.3 ±0.4 ^d
ZA1	86.6 ±0.8 ^b	0.4 ±0.2 ^e	21.4 ±0.9 ^f
ZA3	78.8 ±0.8 ^c	3.3 ±0.4 ^{cd}	32.1 ±1.1 ^d
ZA6	71.9 ±1.1 ^d	6.1 ±0.6 ^a	38.6 ±1.4 ^c
ZW1	85.9 ±1.1 ^b	-0.3 ±0.4 ^f	26.9 ±1.9 ^e
ZW3	78.4 ±0.9 ^c	3.9 ±0.5 ^c	42.1 ±1.3 ^b
ZW6	72.1 ±0.8 ^d	7.5 ±0.5 ^b	48.4 ±0.7 ^a
P	<.001	<.001	<.001

CZ, precolored zirconia; SD, standard deviation; Z, unshaded zirconia (control); ZA1, acid-based coloring liquid, 1 application; ZA3, acid-based coloring liquid, 3 applications; ZA6, acid-based coloring liquid, 6 applications; ZW1, aqueous coloring liquid, 1 application; ZW3, aqueous coloring liquid, 3 applications; ZW6, aqueous coloring liquid, 6 applications. Superscript letters for each column indicate mean values significantly different between groups (P<.001).

blue, (+) indicates yellow, (0) indicates gray, and (-) indicates blue.¹⁷

The data were analyzed using statistical software (IBM SPSS Statistics, v22.0; IBM Corp). A 1-way ANOVA was used to identify differences among all the groups. A 2-way ANOVA was used to evaluate the effect of the type of coloring liquid and number of coloring liquid applications on the color of zirconia. Multiple comparisons were conducted using the Scheffé test to detect statistically significant differences (α=.001).

RESULTS

As outlined in Table 2, significant differences were observed in the CIE L*, a*, and b* values for the different zirconia specimen groups (P<.001). The highest CIE L* value was observed for the control group (unshaded zirconia specimen), which also gave the lowest CIE a* and b* values.

To analyze the differences in the CIE L*, a*, and b* values of the treated specimens for different coloring liquids and number of applications, 2-way ANOVA was performed (Table 3). The CIE a* and b* values were significantly higher for the aqueous coloring liquid group than for the acid-based coloring liquid group (P<.001). This result indicated that the aqueous coloring liquid group exhibited a greater redness/yellowness than the acid-based coloring liquid group. In addition, the similar CIE L* values of the different coloring liquids (P=.25) indicated a comparable brightness for both the groups.

The CIE L* values were significantly lower and the CIE a* and b* values were significantly higher in the aqueous coloring liquid and acid-based coloring liquid groups on increasing the number of applications (P<.001), thereby indicating that the application of coloring to zirconia reduces the material brightness and gives redness and yellowness.

The interaction effect between the type of coloring liquid and the number of coloring liquid applications was

Table 3. CIE L*, a*, b* values of zirconia specimens as analyzed by 2-way ANOVA (±SD)

	No. of Coloring Liquid Application	Acid-Based Coloring Liquid (ZA)	Aqueous Coloring Liquid (ZW)	P
L*	1 ^A	86.6 ±0.8	85.9 ±1.1	α<.001
	3 ^B	78.8 ±0.8	78.4 ±0.9	β=.250
	6 ^C	71.9 ±1.1	72.1 ±0.8	α*β=.324
a*	1 ^A	0.4 ±0.2	-0.3 ±0.4	α<.001
	3 ^B	3.3 ±0.4	3.9 ±0.5	β<.001
	6 ^C	6.1 ±0.6	7.5 ±0.5	α*β<.001
b*	1 ^A	21.4 ±0.9	26.9 ±1.9	α<.001
	3 ^B	32.1 ±1.1	42.1 ±1.3	β<.001
	6 ^C	38.6 ±1.4	48.4 ±0.7	α*β<.001

SD, standard deviation; ZA, acid-based coloring liquid; ZW, aqueous coloring liquid; α, number of coloring liquid application; β, coloring liquid. Superscript letters for each row indicate mean values significantly different between groups (P<.001).

Table 4. ΔE* value among zirconia specimen groups

Group	Z	CZ	ZA1	ZA3	ZA6	ZW1	ZW3	ZW6
Z	—	29.0	17.3	30.7	40.2	22.6	39.8	48.7
CZ	29.0	—	12.2	6.9	15.0	7.1	11.4	20.8
ZA1	17.3	12.2	—	13.6	23.3	5.6	22.6	31.5
ZA3	30.7	6.9	13.6	—	17.9	9.6	12.7	17.9
ZA6	40.2	15.0	23.3	17.9	—	19.3	7.8	9.9
ZW1	22.6	7.1	5.6	9.6	19.3	—	17.5	26.8
ZW3	39.8	11.4	22.6	12.7	7.8	17.5	—	9.6
ZW6	48.7	20.8	31.5	17.9	9.9	26.8	9.6	—

CZ, precolored zirconia; SD, standard deviation; Z, unshaded zirconia (control); ZA1, acid-based coloring liquid, 1 application; ZA3, acid-based coloring liquid, 3 applications; ZA6, acid-based coloring liquid, 6 applications; ZW1, aqueous coloring liquid, 1 application; ZW3, aqueous coloring liquid, 3 applications; ZW6, aqueous coloring liquid, 6 applications.

significant for the CIE a* and b* values (P<.001). The CIE a* value increased with the number of coloring liquid applications in all the coloring liquids, but the CIE a* value increased more than the acidic coloring liquid when the aqueous coloring liquid was applied 3 or more times. The CIE b* value increased with the number of coloring liquid applications in all the coloring liquid, but the CIE b* value of the aqueous coloring liquid increased more than that of the acidic-based coloring liquid when applied 3 times.

The results obtained after the analysis of the color differences (ΔE*) are shown in Table 4. The color difference ranged from 5.6 to 48.7ΔE. The least ΔE* value was 5.6 between ZA1 and ZW1 groups. The highest ΔE* value was 48.7 between Z and ZW6 groups. The color difference between the control group (Z, unshaded zirconia specimen) and the group containing zirconia treated with coloring liquid (ZW and ZA) increased as the number of applications increased (ΔE>3.7). The precolored zirconia group gave the least color difference in the case of the ZA3 group (6.9) and ZW1 group (7.2) (ΔE>3.7). With an equal number of applications, the color difference between the ZA1 and ZW1 specimens

was 5.6, which is the minimum value recorded. In contrast, the maximum difference was observed between the ZA3 and ZW3 groups (12.7). This indicates that the largest difference was obtained after 3 applications of the coloring liquid. In all groups, the ΔE^* value was greater than 3.7, and a visible color difference was apparent.

DISCUSSION

The null hypothesis that the color of zirconia is not affected by the type of coloring liquid used was partially adopted. As indicated previously, although the CIE L^* values remained constant for the various coloring liquids, the values of CIE a^* and b^* were higher for the aqueous coloring liquid group than for the acid-based coloring liquid group. These results confirm that the 2 groups exhibited an equal brightness, but the aqueous coloring liquid produced a greater redness and yellowness than the acid-based coloring liquid. In addition, the ΔE^* value, which represents the color difference between different coloring liquids, gave the lowest value of 5.6 after a single application and the highest value of 12.7 after 3 applications. Interestingly, 6 applications gave an ΔE^* value of 9.9, which was lower than that recorded after 3 applications.

Regardless of the number of applications, the ΔE^* value was influenced mainly by the CIE b^* value, which was an indicator of the blue–yellow system. This indicates that the aqueous coloring liquid exhibited a greater yellowness compared with the acid-based coloring liquid, likely because of the different metal salts used and the different pH values of the coloring liquids. This difference should be considered when deciding on the type of coloring liquid.¹⁵

In this study, the range of ΔE^* values was 5.6 to 48.7, which was higher than the visible color difference of 3.7. In a study by Kim and Kim¹⁶ on the effect of the number of coloring liquid applications on the optical properties of monolithic zirconia, the range of ΔE^* values was found to be 1.3 to 15.7. In a study by Kim et al¹⁵ on the effect of coloring agent on the color of zirconia, the range of ΔE^* values was from 0.87 to 9.43. The results of these studies showed a small color difference compared with the results of this study, which was a difference according to the coloring technique of the operator.

The null hypothesis that the color of zirconia is not affected by the number of applications was rejected. As the number of applications increased, the CIE L^* values decreased, and the CIE a^* and b^* values increased for all groups. This indicates that irrespective of the type of coloring liquid used, an increase in the number of applications decreased the specimen lightness with redness or yellowness. For comparison, Kim et al^{15,16} reported that the CIE L^* and opalescence parameter values decreased upon increasing the number of applications,

whereas the CIE b^* values increased. This result is consistent with the results of the present study.

The null hypothesis that there is no interaction effect between the type of coloring liquid and the number of coloring liquid applications was partially adopted. The CIE a^* value increased with the number of coloring liquid applications in all the coloring liquids, but the CIE a^* value increased in the aqueous coloring liquid more than the acidic-based coloring liquid when the coloring liquid was applied 3 or more times. The CIE b^* value increased with the number of coloring liquid applications in all the coloring liquid, but the CIE b^* value increased in the aqueous coloring liquid more than the acidic coloring liquid when the coloring liquid was applied 3 times. Therefore, the ΔE^* between the 2 colorants appeared to be higher at 3 times (ΔE : 12.7) than when the coloring agent was applied 6 times (ΔE : 9.9).

The results of this study showed that the lowest color difference, ΔE^* , between the precolored zirconia (CZ) group and the acid-based coloring liquid group was obtained after 3 applications of the acid-based coloring liquid, while the lowest difference between the CZ group and the aqueous coloring liquid group was obtained after a single application of the aqueous coloring liquid. Although the CIE L^* values were comparable with those of the CZ group after a single application of the acid-based coloring liquid, large color differences were observed between different groups because of the CIE b^* value. Upon 3 applications of the acid-based coloring liquid, the CIE L^* values decreased slightly and the CIE a^* and b^* values increased to values similar to those of the CZ group. A ΔE^* value of 6.9 was, therefore, obtained between the CZ group and the ZA3 group, which was lower than that calculated after a single application of the acid-based coloring liquid.

Upon a single application of the aqueous coloring agent, the color difference, ΔE^* , between this group and the CZ group gave the lowest value of 7.1. Comparable CIE L^* values were also recorded, although the CIE a^* and b^* values differed slightly. This result means that when a colorant is applied to zirconia, it is necessary to apply a coloring agent once for the aqueous coloring agent and to apply the coloring agent 3 times for the acidic-based coloring agent to obtain the same color as the precolored zirconia.

The natural color of teeth has the CIE L^* values ranging from 78.0 to 64.3, the CIE a^* values from 2.8 to 2.6, and the CIE b^* values from 25.7 to 9.8.¹⁸ In this study, the CIE L^* , a^* and b^* values of the precolored zirconia (CZ) group were 85.5, 2.9, and 33.3, respectively, which indicated an increased brightness and a greater yellowness compared with the natural tooth color.

Three applications of aqueous coloring liquid (CIE L^* , a^* , and b^* : 78.4, 3.9, and 42.1) and acid-based coloring liquid (CIE L^* , a^* , and b^* : 78.8, 3.3, and 32.1) were most

similar to the natural color of teeth but had increased yellowness because of the high CIE b^* value, and the aqueous coloring liquid exhibited greater yellowness. This should be considered in clinical practice.

In this study, the Zirkonzahn aqueous coloring liquid was used, and the obtained CIE a^* and b^* values for the aqueous coloring liquid group were higher than those of the acid-based coloring liquid group. Based on the previous studies,^{13,14} it was considered that higher CIE a^* and b^* values were obtained for the red and yellow series as the aqueous coloring liquids contained greater iron content than the acid-based coloring liquids.

The limitations of this study include that a single aqueous colorant and an acid-based colorant were used for comparisons. Therefore, it is necessary to study the effect of various coloring agents on the coloring of zirconia. In addition, the flexural strength and compressive strength of zirconia restorations should be studied to understand the effects of various coloring agents on the mechanical properties of zirconia. It is also necessary to examine any changes in color caused by polishing and occlusion adjustment of the restoration.

CONCLUSIONS

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

1. The CIE a^* and b^* values of the aqueous coloring liquid group were statistically higher than those of the group treated with an acid-based coloring liquid ($P < .001$). This indicates that treatment with aqueous coloring liquid on zirconia produced a greater redness or yellowness compared with treatment with acid-based coloring liquid.
2. The decrease in the CIE L^* values and the increase in the CIE a^* and b^* values were statistically significant on increasing the number of color applications in all groups ($P < .001$). This indicates the coloring of zirconia lowers its brightness and imparts a red/yellow hue.

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