



Effect of different coloring liquids on the flexural strength of multilayered zirconia

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PURPOSE. The purpose of this study was to evaluate the effect of two coloring liquids (aqueous and acid-based coloring liquids) and the position of multilayered zirconia on the flexural strength of multilayered zirconia.

MATERIALS AND METHODS. The multilayered zirconia specimens were divided into upper and lower positions. The specimens were divided into three subgroups (n=10): non-shaded, acid-based coloring liquid, and aqueous coloring liquid. The specimens were cut using a milling machine and were immersed in either an acid-based coloring liquid or aqueous coloring liquid 2 times for 5 seconds. The specimens were sintered in a sintering furnace according to the manufacturer's introduction. The flexural strength of the specimen was measured using a universal testing machine and the surface of the specimen was observed using a field emission scanning electron microscope. **RESULTS.** The flexural strength of multilayered zirconia was 400 - 500 MPa. There was no statistically significant difference among all groups ($P>.05$). The flexural strength of the multilayered zirconia was not influenced by the kind of coloring liquid used ($P>.05$). The flexural strength of the multilayered zirconia colored with the coloring liquids was not influenced by its position ($P>.05$). **CONCLUSION.** The different coloring liquid application did not affect the flexural strength of multilayered zirconia of all positions. [*J Adv Prosthodont* 2019;11:209-14]

KEYWORDS: Multilayered zirconia; Coloring liquid; Flexural strength

INTRODUCTION

Among various dental materials, zirconia has recently attracted much attention because of its high strength and excellent mechanical properties. In addition, zirconia has been applied to various fields of dentistry due to the development of computer-aided design and computer-aided manufacturing (CAD/CAM) systems. The initially used white zirconia blocks were stabilized tetragonal zirconia prepared by adding 3 mol% yttrium to pure zirconia powder.^{1,2} White zirconia has been used only as a core for veneer

ceramics because it could not reproduce natural hues with high opacity and low saturation.³ However, failure due to the thermal expansion coefficient difference between the two materials has often been reported.^{4,6} As an alternative to this, monolithic or full-contour zirconia restorations have been developed and commercialized.⁷

The monolithic zirconia restorations showed a high strength with a thin thickness, but have been not used in the anterior region but only in the posterior crown due to their optical properties.⁷ However, several coloring techniques^{8,9} and a variety of zirconia blocks such as a high-translucency monolithic zirconia block^{10,11} and a multilayered zirconia block^{12,13} have been introduced and the applications of monolithic zirconia have been expanded.¹⁴

Methods of imparting color to zirconia blocks include additive techniques and painting techniques.^{15,16} In the additive techniques, metal oxides or other materials capable of reproducing a color are added in the process of forming the zirconia powder, and after pressing zirconia powder, the resultant colored zirconia block is presintered. The painting techniques are a method of directly dipping zirconia into coloring liquids, or applying coloring liquids to zirconia with a brush to obtain a desired color. The coloring liquids used in

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the painting techniques include acid-based coloring liquids that contain a strong aqueous hydrogen chloride solution, which impart the color via an acid-based reaction, and aqueous coloring liquids that do not contain an acid component.¹⁷

Transparency is an important criterion in the selection of materials and is the main factor affecting esthetics.^{11,12} High-transparency zirconia has been introduced with properties superior to those of the opaque monolithic zirconia. High-transparency zirconia blocks have been produced by increasing the content of the transparent yttria powder to more than 7 wt% or by decreasing the particle size.¹⁸ As the content of yttria increases, the cubic phase in the zirconia increases, resulting in the higher translucency.^{19,20} Different manufacturers prepare high-transparency zirconia blocks using different powder blending ratio, sintering temperature, molding method, and other factors.¹¹ The development of high-transparency zirconia blocks has extended the application of single zirconia restorations to both the anterior and posterior regions.¹⁹

Recently, various multilayered zirconia materials have been introduced to improve the color of monolithic zirconia. The multilayered zirconia block has transparency and color similar to those of natural tooth, and has various color layers corresponding to the enamel and dentin in the block itself. It has various color layers corresponding to the incisal layer to the cervical layer similar to the natural tooth.²⁰ There are two methods for manufacturing a multilayered zirconia block: immersing in coloring liquids and adjusting the ratio of constituent components' contents.

The method of immersing in coloring liquids includes two techniques: manufacturing a zirconia block using a coloring liquid with adjustable brightness/saturation²¹ and manufacturing a zirconia block by varying the absorption rate of the coloring solution by varying the particle size of the zirconia layer.²² Further, the content ratio of constituent components can be adjusted via four methods: manufacturing a multilayered zirconia block by adjusting the content of yttrium oxide²³; manufacturing a multilayered zirconia block by adjusting white zirconia-based powder/colored zirconia-based powder content²⁴; manufacturing a block having various light transmittance values, colors, and strengths by varying the content of zirconia powder, rare earth raw material (yttrium oxide powder), and colorant powder²⁵; manufacturing a zirconia block with improved light transmittance by selectively controlling the content ratio of yttria and the color component and by adding alumina.²⁶

Previous studies have reported that coloring liquids affected the mechanical properties of zirconia.^{9,27-29} Hjerpe *et al.*²⁷ reported that dipping white zirconia in a coloring liquid had a negative effect on the flexural strength of the specimen. Orhun⁹ reported that prolonged immersion of zirconia in a coloring liquid negatively affected the fracture load of the monolithic zirconia. Nam and Park²⁹ reported that applying aqueous and acid-based coloring liquids to monolithic zirconia resulted in a decrease in the hardness value only in the case of specimens painted with acid-based coloring liquids. Ban *et al.*²⁸ reported that the flexural

strength and fracture toughness of zirconia, Cercon base (white zirconia), ZENOSTAR Pure and Zirkozahn Prettau (monolithic zirconia), were lowered when Er and Nd ions were included in the coloring liquids. On the other hand, Sedda *et al.*³⁰ reported that the coloring liquids had no effect on the flexural strength of zirconia, In-Ceram YZ (monolithic zirconia) and In-Ceram YZ Color LL1p (preshaded monolithic zirconia). In addition, Kong *et al.*³¹ reported that there was almost no difference in flexural strength between specimens formed by immersing shaded high-transparency zirconia in two acid-based coloring liquids with different pH. However, to our knowledge, no study has considered the effects of the coloring liquid on multilayered zirconia.

Further, previous studies have shown that the zirconia strength decreases with increasing transparency.^{3,10,32,33} In the studies of Muñoz *et al.*³³ and Carrabba *et al.*,¹⁰ there was a negative correlation between transparency and flexural strength, and with increasing yttria content in the zirconia block, the flexural strength decreased. Pereira *et al.*³ also reported that the higher the transparency, the lower the flexural strength of multilayered zirconia. In addition, Park³² compared the fracture strengths of multilayered zirconia, nonpreshaded monolithic zirconia, and preshaded monolithic zirconia. Although the strength of multilayered zirconia was the lowest, it was reported that sufficient flexural strength was obtained with acceptable occlusal force at the posterior portion.

However, there is no study on the effect of coloring liquids on the strength of multilayered zirconia. The purpose of this study was to evaluate the effect of the two coloring liquids (aqueous and acid-based coloring liquids) and the position of multilayered zirconia on the flexural strength of multilayered zirconia. The first null hypothesis was that the coloring liquids do not affect the flexural strength of the multilayered zirconia. The second null hypothesis was that the flexural strength of multilayered zirconia does not vary depending on the position.

MATERIALS AND METHODS

In this study, specimens were fabricated using multilayered zirconia blocks A3 (3M Lava Esthetic, 3M Deutschland GmbH, Germany, Lot No. 669633) with 5 mol% yttria as stabilizer. The size of the multilayered zirconia specimen used in the flexural strength test was selected according to the ISO 6872: 2015 standard.³⁴ The specimens were designed using CAD software (Auto CAD 2006, Autodesk, Mill Valley, CA, USA) and specimens were cut using a milling machine (Roland DWX-52D, Roland, Hamamatsu, Japan) by considering the shrinkage percentage of the green-stage zirconia blocks. Specimens were prepared by dividing the multilayered zirconia block into upper and lower layers. The upper layer is where the incisal part of the restorations is located, and the lower layer is where the cervical part of the restorations is located (Fig. 1). The size of all specimens was measured using vernier calipers (Digimatic Caliper, Mitutoyo, Kawasaki, Japan) and polished with #1,000 grit SiC paper

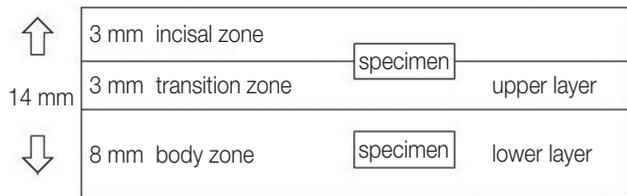


Fig. 1. The upper and lower layer of multilayered zirconia.

Table 1. Materials used (n = 10)

Materials	Coloring liquid	Zirconia position	Group code	
	-	Upper	ZU	Z
		Lower	ZL	
3M Lava Esthetic	Acid-based	Upper	KU	K
		Lower	KL	
	Aqueous	Upper	AU	A
		Lower	AL	

ZU, non-shaded, upper (control); ZL, non-shaded, lower (control); KU, acid-based coloring liquid, upper; KL, acid-based coloring liquid, lower; AU, aqueous coloring liquid, upper; AL, aqueous coloring liquid, lower.

to a final size of $25 \times 4.2 \times 1.2$ mm. A total of 60 specimens were divided into 6 groups (n = 10) (Table 1). The specimens were immersed in A3 acid-based (Ko's Liquid, Kuwotech, Gwangju, Korea) and aqueous coloring liquids (Colour Liquid for Prettau® Aquarell, Zirkozahn, Italy Lot No. CB4484) 2 times for 5 seconds. After immersion, all specimens were dried in an oven at 150°C for 15 minutes. The dried specimens were sintered in a sintering furnace (LHT 02/17 / LB, Nabetherm, Lienthal, Germany) according to the schedule specified by the manufacturer. The temperature was raised at a rate of 20°C/min to 800°C and then at a rate of 10°C/min to 1,500°C. The specimens were sintered for 2 hours at 1,500°C, after which the temperature decreased at a rate of 15°C/min to 800°C and then at a rate of 20°C/min to 250°C and were slowly cooled to room temperature. The specimens of the control group were sintered without being immersed in the coloring liquids.

A universal tester (STM-5; United Calibration Corporation, Fullerton, CA, USA) was used to measure the flexural strength of the multilayered zirconia specimens. The specimens were placed at the center of the support with a width of 20 mm and the load was applied vertically at the center of the specimen with a crosshead speed of 1 mm/min until the specimen was fractured. The average value was considered to be the flexural strength of the specimens. The maximum load (fracture load) was recorded in Newton, and the

flexural strength (σ) was calculated as follows in megapascal:

$$\sigma = 3Pl / 2wb^2,$$

where P is the applied load (in N), l is the test interval (in mm), w is the width of specimen (in mm), and b is the thickness of the specimen (in mm).

In order to observe the effect of the coloring liquids, each specimen of 6 groups was immersed in an 80% alcohol solution and ultrasonically cleaned for 3 minutes. The surface was coated with osmium as a Pure Osmium Coster (NEOC, Meiwafohis, Japan), and then, the surface was analyzed using a field emission scanning electron microscope (JSM-6700F, JEOL, Tokyo, Japan). A one way analysis of variance (ANOVA) and a two way ANOVA were performed using the software (SPSS v.25.0, SPSS Inc., Chicago, IL, USA) to test the statistical significance of the test results. Post-test Scheffe test was performed for multiple comparisons ($\alpha = .05$).

RESULTS

Table 2 shows the results of the flexural strength of the multilayered zirconia specimens according to the application of the coloring liquids. The flexural strength of multilayered zirconia was 400 - 500 MPa. There was no statistically significant difference among all groups ($P > .05$). The flexural strength of the multilayered zirconia did not show significant differences among the coloring liquids ($P > .05$) (Table 3). The flexural strength of the multilayered zirconia did not show significant differences between the positions ($P > .05$) (Table 3).

Further, the changes in the multilayered zirconia specimens as a result of the coloring liquid application were observed by field scanning electron microscopy (Fig. 2). The microstructure of the zirconia specimen was isotropic. In the specimen to which the aqueous coloring liquid was applied, a coloring liquid was absorbed and a surface was similar to that of the control. On the other hand, it was observed that the coloring liquid remained partially on the surface of the specimen to which the acid-based coloring

Table 2. Mean and standard deviation of flexural strength of zirconia specimens (n = 10)

Group	Mean \pm SD (MPa)
ZU	433.38 \pm 59.30 ^a
ZL	443.03 \pm 38.33 ^a
KU	424.12 \pm 42.76 ^a
KL	433.94 \pm 37.60 ^a
AU	444.81 \pm 40.59 ^a
AL	461.62 \pm 59.57 ^a

ZU, non-shaded, upper (control); ZL, non-shaded, lower (control); KU, acid-based coloring liquid, upper; KL, acid-based coloring liquid, lower; AU, aqueous coloring liquid, upper; AL, aqueous coloring liquid, lower. The same superscript letters in the same column show not significant differences ($P > .05$).

Table 3. Flexural strength test of specimens, as analyzed by two-way ANOVA (\pm SD)

Position	Non-shaded (Z) ^A	Acid-based coloring liquid (K) ^A	Aqueous coloring liquid (A) ^A	P
Upper ^a	433.38 \pm 59.30	424.12 \pm 42.76	444.81 \pm 40.59	$\alpha = 0.272$ $\beta = 0.326$
Lower ^a	443.03 \pm 38.33	433.94 \pm 37.60	461.62 \pm 59.57	$\alpha \times \beta = 0.963$

α , coloring liquid; β , position; Z, non shaded; K, acid-based coloring liquid; A, aqueous coloring liquid. The same superscript letters in the same column show not significant differences ($P > .05$).

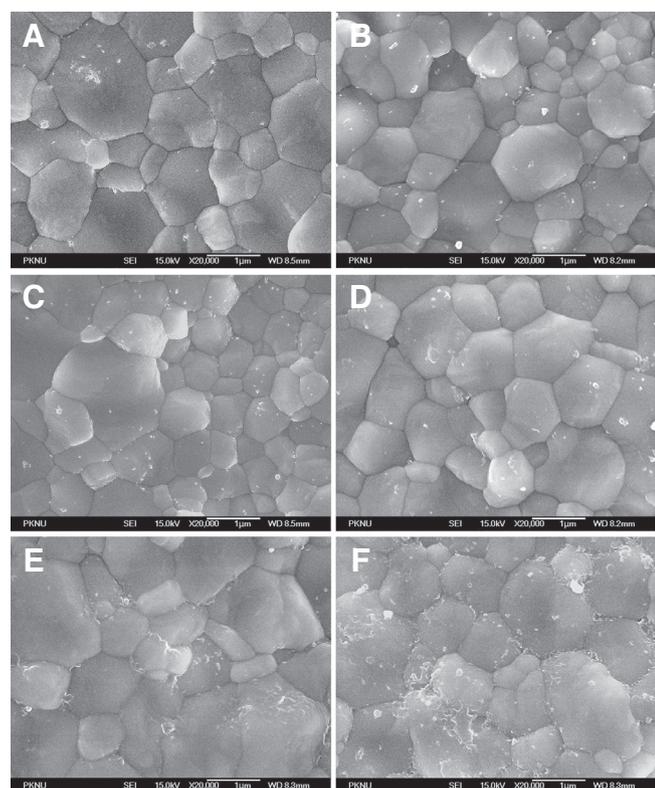


Fig. 2. Field emission scanning electron micrograph images of multilayered zirconia specimens (3M Lava Esthetic). (A) ZU, non-shaded, upper; (B) ZL, non-shaded, lower; (C) KU, acid-based coloring liquid, upper; (D) KL, acid-based coloring liquid, lower; (E) AU, aqueous coloring liquid, upper; (F) AL, aqueous coloring liquid, lower. (Original magnification, $\times 20,000$)

liquid was applied. Further, no significant change in the grain size with the application of a coloring liquid was observed.

DISCUSSION

There was no statistically significant difference in the flexural strength among the coloring liquids. Therefore, the first null hypothesis that coloring liquid applications would not affect the flexural strength of zirconia was confirmed.

There was no statistically significant difference in the flexural strength between the positions, so a second null hypothesis that zirconia flexural strength did not differ

Table 4. Clinical recommendation proposed by ISO 6872:2015 dental ceramics

Class Recommended Clinical Indications	Flexural strength minimum (MPa)
Ceramic for coverage of a metal framework or a ceramic substructure	50
Monolithic ceramic for single-unit anterior prostheses, veneers, inlays, or onlays	
Monolithic ceramic for single-unit, anterior or posterior prostheses adhesively cemented	
Partially or full covered substructure ceramic for single-unit anterior or posterior prostheses adhesively cemented	100
Monolithic ceramic for single-unit anterior or posterior prostheses and three-unit prostheses not involving molar restoration adhesively or non-adhesively cemented	
Partially or fully covered substructure for single-unit anterior or posterior prostheses and for three-unit prostheses not involving molar restoration adhesively or non-adhesively cemented	300
Monolithic ceramic for three-unit prostheses involving molar restoration.	
Partially or fully covered substructure for three-unit prostheses involving molar restoration.	500
Monolithic ceramic for prostheses involving partially or fully covered substructure for four or more units or fully covered substructure for prostheses involving four or more units.	800

according to the position was confirmed. In all specimens, the flexural strength was between 400 and 500 MPa, which is higher than 300 MPa, so according to ISO 6872:2015 (Table 4),³⁴ the flexural strength is acceptable for the monolithic ceramic for single-unit anterior or posterior prostheses and three-unit prostheses not involving molar restoration.

Sedda *et al.*³⁰ compared the flexural strengths according to the coloring techniques: monolithic zirconia blocks, pre-shaded monolithic zirconia blocks, and zirconia blocks immersed in coloring liquid. Their results showed that there was no significant difference among the groups and that the coloring liquid did not affect the flexural strength of the zir-

conia. Further, Kong *et al.*³¹ compared three-point flexural strength of the pre-shaded high-translucency zirconia treated with coloring liquids having a pH of 3.8 and 5.6. The specimens immersed in the pH of 5.6 liquid showed a higher flexural strength than that treated with the other liquid, but no significant difference in the flexural strength was observed. The results of our study are consistent with the findings of Sedda *et al.*³⁰ and Kong *et al.*³¹ in that the coloring liquid did not affect the flexural strength of zirconia.

On the other hand, Orhun⁹ and Hjerpe *et al.*²⁷ reported that coloring liquid application and increasing coloring time decreased the strength of zirconia. Ban *et al.*²⁸ reported that most of the crystal phase of the zirconia colored with coloring liquid containing Er and Nd ions was cubic, resulting in lower flexural strength and fracture toughness. Nam and Park²⁹ reported that the hardness of the monolithic zirconia treated with the acid-based coloring liquid was significantly lower than that of the zirconia treated with the aqueous coloring liquid. It was also observed that the former had a surface covered with the coloring liquid component that was not completely absorbed between the crystal grains. In this study, it was also observed that in the specimens treated with the acid-based coloring liquid (with the low flexural strength), the coloring liquid remained on the surface and was not completely absorbed into the crystal grains of zirconia.

Pereira *et al.*³ and Park³² reported that the grain size affected the strength of multilayered zirconia. Park³² compared the fracture strengths of multilayered zirconia, non-pre-shaded monolithic zirconia, and pre-shaded monolithic zirconia. Non-shaded monolithic zirconia showed the highest fracture strength and multilayered zirconia showed the lowest strength. This was because the grains of untreated zirconia were small and this had a positive effect on the fracture strength. Pereira *et al.*³ studied the flexural strength of multilayered zirconia blocks with different transparency: ML (high-translucent), STML (super-translucent), UTML (ultra-translucent). ML with the lowest transparency showed the highest strength of 889 MPa, STML showed 507 MPa, and UTML with the highest transparency showed the lowest strength of 470.2 MPa. STML and UTML had an yttrium oxide content of 8.15% and 9.32%, and these showed fully stabilized crystal structures in the tetragonal and cubic phases, while ML contained 5.66 wt% yttrium oxide and was mainly composed of tetragonal crystals. ML had the highest strength because zirconia grains were denser than those in UTML and STML. In this study, the grain size did not change with the coloring liquid applications, which seems to be related to the lack of significant difference in the flexural strength.

Zirconia exists in three phases depending on the temperature. As the temperature increases, it has a monoclinic phase up to 1,170°C at room temperature, a tetragonal phase up to 2,370°C, and a cubic phase above 2,370°C. However, when zirconia is allowed to remain at a low temperature for a long period of time in moisture, or when cracks are generated by external stimuli during the cooling process, zirconia mechanical properties begin to deteriorate. At this time, the tetragonal to monoclinic phase transition

occurs, and yttria is added as a stabilizer to suppress the phase transition.³⁵

Previous studies have reported that the strength of zirconia blocks containing yttria are much lower.^{10,33} Muñoz *et al.*³³ reported that as zirconia contains more yttria, grain size was larger and transparency was higher, but biaxial flexural strength decreased. Carrabba *et al.*¹⁰ also compared the three-point flexural strength of zirconia containing 3 mol% and 5.5 mol% yttria. The latter presented a lower flexural strength. That is, as the content of yttria was increased, the flexural strength of zirconia decreased. Denry *et al.*¹ reported that the flexural strength of white zirconia containing 3 mol% yttria as a stabilizer (3Y-TZP) was 800 - 1,000 MPa. In the case of conventional white 3Y-TZP, 3 mol% yttria was added as a stabilizer, but the multilayered zirconia used in this study was composed of 5 mol% yttria. In this study, the flexural strength of multilayered zirconia was 400 - 500 MPa, which was similar to that reported by Pereira *et al.*³ This decrease in the flexural strength was thought to be due to an increase in the yttria content.^{10,33}

The color of the coloring liquid and multilayered zirconia used in this study was A3, the most commonly used coloring liquid in clinical practice. In preliminary experiments, multilayered zirconia specimens were divided into upper, middle, and lower positions because the multilayered zirconia block used in this study consists of 3 layers: incisal zone, transition zone, body zone. There was no difference in flexural strength between the upper and middle positions. In this study, multilayered zirconia specimens were divided into upper and lower parts. In this study, the effect of the coloring liquid and zirconia position on the flexural strength of one kind of multilayered zirconia was investigated. However, it is necessary to investigate mechanical properties such as compressive strength and hardness through various strength tests as well as to analyze the flexural strength of various multilayered zirconia blocks. In addition, the influence of the components on the zirconia strength by analyzing the components of the coloring liquids in multilayered zirconia has to be investigated. Further, analysis of the penetration depth of the coloring liquids in the multilayered zirconia and color and transparency change due to the coloring liquid application is also necessary.

CONCLUSION

Within the limits of this study, the following conclusions were obtained. The flexural strength of the multilayered zirconia specimen according to the coloring liquid application was 400 - 500 MPa to be acceptable for the monolithic ceramic for single-unit anterior or posterior prostheses and three-unit prostheses not involving molar restoration. The coloring liquid application did not reduce the flexural strength of multilayered zirconia of all position.

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