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Influence of coloring procedure on flexural resistance of zirconia blocks

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ABSTRACT

Statement of problem. The white color of zirconia limits the possibility of a natural-looking restoration. Industrially precolored blocks and green-stage coloring liquids are used to obtain shaded zirconia. Several authors have reported that pigments may negatively affect the mechanical characteristics of the material.

Purpose. The purpose of this in vitro study was to investigate the effect of a recently marketed coloring system on the flexural strength (σ), Weibull characteristic strength (σ_0), and Weibull modulus (m) of a zirconia for computer-aided design/computer-aided manufacturing (CAD/CAM) systems.

Material and methods. Noncolored white blocks (In-Ceram YZ), industrially precolored blocks (In-Ceram YZ col. LL1p), and green-stage infiltration-shaded blocks in 4 colors (In-Ceram YZ Coloring Liquids) were tested. Industrially precolored specimens (n=30) were assigned to the PREC group. Noncolored specimens were divided into 5 groups (n=30). Group CNTR was not treated (control). Groups INF-N, INF-L, INF-M, and INF-I were subject to green-stage infiltration (neutral, light, medium, and intense, respectively). Specimens were tested in a 3-point bending apparatus following ISO 6872:2008 specifications. Data were analyzed by 1-way ANOVA.

Results. The calculated flexural strengths (MPa) were CNTR 1232.56 ±103.91; PREC 1215.16 ±141.42; INF-N 1280.72 ±160.10; INF-L 1218.92 ±78.63; INF-M 1153.71 ±176.80; and INF-I 1248.46 ±111.73. No statistically significant differences were found among the tested groups (*P*=.195).

Conclusions. Unlike what has been reported for other combinations of zirconia and coloring liquids, the coloring technique tested in the present study did not negatively influence the flexural characteristics of the tested zirconia. (J Prosthet Dent 2015;114:98-102)

Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP), commonly referred to as zirconia, is gaining popularity in dentistry because of its biological, mechanical, and esthetic properties. Zirconia has been found to have higher flexural strength and fracture toughness than any other ceramic materials used for dentistry.[1-3] These mechanical properties derive from the mechanism called transformation toughening.[4,5] Pure zirconium oxide exists in 3 crystallographic forms: monoclinic, tetragonal, and cubic.[6,7] However, only the monoclinic form is stable at room temperature, and therefore stabilizers such as yttria (Y_2O_3) or ceria (CeO_2) are added to maintain the zirconia in tetragonal form.[8,9] According to Kelly and Denry,[10] the simplest, most commonly understood toughening mechanism concept involves crack tip shielding (from the applied stress) by the compressive dilatational stress associated with transformation. In fact, more complex factors, such as hydrostatic components, microstructure of the transformed material, R-curve behavior, and shear versus dilatational stresses, are involved in the zirconia toughening mechanism.[9,10]

Zirconia is currently used to fabricate fixed dental prosthesis frameworks veneered with traditional feldspathic porcelain, monolithic crowns, and implant abutments.[11-13] The Y-TZP is used with different

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Clinical Implications

For the combination of zirconia and coloring liquids tested in the present study, green-stage infiltration with coloring liquids can be used to shade zirconia without altering its flexural strength.

computer-aided design/computer-aided manufacturing (CAD/CAM) techniques and is most commonly milled at the green-stage form. In fact, after a dense-sintering process, this material achieves an extreme hardness, and the milling process would be more difficult.[14,15] One of the most important advantages of zirconia ceramic restorations over the traditional metal ceramic restorations is their more esthetic appearance. Unlike metal frameworks, zirconia has the esthetic advantage of not being completely opaque, depending on its microstructure and thickness, supporting the current definition of zirconia as a semitranslucent core material.[16] Moreover, the white aspect can mask the frame collar, which is responsible for the unesthetic black line effects on metal ceramic restorations.[17] Notwithstanding these advantages, the color of zirconia is white,[18] and obtaining a natural tooth color may be difficult to achieve even after veneering because the color of the framework affects the final color of the restoration.[19,20] For these reasons, using a shaded zirconia may facilitate a more natural appearance.

Three main approaches to obtaining a shaded zirconia are currently available for dental purposes: (1) metal oxides can be mixed with the starting ZrO_2 powder at the production stage to obtain precolored green-stage blocks; (2) before sintering, green-stage frameworks can be infiltrated with specific coloring liquids; and (3) after sintering, zirconia can be painted with liners that require firing in a traditional dental ceramic furnace. The last method, however, has been considered a weak link of the zirconia veneering procedure for some commercial systems and has been reported to be one of the possible explanations for zirconia veneering ceramic delamination.[21] However, doping with coloring substances, usually metal oxides, has been reported to decrease the mechanical properties of the zirconia.[22,23]

In industrial manufacturing, there is a growing trend for colored zirconia to be used as a replacement for metallic decorative materials used as ornament workings, in optical components, or as structural parts.[24-28] This trend has encouraged the investigation of different coloring techniques for zirconia, such as heterogeneous nucleation and injection molding techniques, to produce a variety of colors.[24-28] The literature relating to the effects of the shading procedure on the mechanical properties of biomedical zirconia is limited.[29-37]

The purpose of this study was to verify whether a recently marketed zirconia coloring liquid would have a negative effect on the mechanical properties of a commercially available zirconia for the CEREC CAD/CAM system. Zirconia colored with coloring liquids was compared to zirconia precolored at the industrial stage and with uncolored zirconia with regard to flexural strength (σ), Weibull characteristic strength (σ_0), and Weibull modulus (m). The tested null hypotheses were that the coloring procedure does not affect the flexural strength, Weibull characteristic strength, or Weibull modulus of the selected zirconia material.

MATERIAL AND METHODS

Two yttria partially stabilized green-stage zirconium dioxide block types, marketed for the CEREC CAD/CAM system (Sirona Dental), were selected for this study: In-Ceram YZ and In-Ceram YZ Color LL1p (Vita). Specimens were prepared according to ISO 6872:2008.[38] The blocks were fixed to a low-speed, water-cooled diamond saw (Low Speed Saw; IsoMet). With the use of a proprietary device, the blocks were first cut longitudinally and then turned 90 degrees clockwise to obtain bar-shaped specimens. To perform an accurate cutting procedure, the speed was maintained below 250 rpm and no extra weight was put on the blocks. Cutting measures were calculated to compensate for the shrinkage induced by dense-sintering. After cutting, 30 precolored and 150 noncolored specimens were obtained. Precolored specimens were assigned to the PREC group; noncolored specimens were assigned to the CNTR group (control). Groups INF-N, INF-L, INF-M, and INF-I were subject to green-stage infiltration (neutral, light, medium, and intense, respectively). Each group consisted of 30 specimens, treated as follow. For the CNTR group, no coloring procedure was performed. The PREC group comprised precolored blocks, industrially shaded by the manufacturer. No further coloring procedure was performed. For the INF-N, INF-L, INF-M, and INF-I groups, green-stage color infiltration with neutral, light, medium, and intense liquids was performed. Specimens at the green stage were dipped with plastic tweezers into the coloring liquid (In-Ceram YZ Coloring Liquid; Vita) for 2 minutes following the manufacturer's instructions. Each group was infiltrated with 1 coloring liquid. Once removed, the excess coloring liquid was gently absorbed with blotting paper.

Sintering was performed according to the manufacturer's instructions (ZYrcomat T; Vita Zahnfabrik). After the sintering process, specimens were wet finished with 600-grit paper until dimensions of 15 ±0.2 mm in length, 4 ±0.2 mm in width, and 1.5 ±0.2 mm in height were obtained. Specimens were then wet polished with 1200- and 2400-grit paper. According to ISO 6872:2008,[38] a

45-degree edge chamfer was made at each of the major edges by keeping the specimens at 45 degrees with the 1200-grit paper disk with a metal rig. A regeneration firing was performed in a ceramic furnace (Vacumat 4000 T; Vita Zahnfabrik) for each group after the finishing procedure, according to the manufacturer's instructions. A 3-point bending test (3PBT) apparatus was prepared. The tip and the supports were made of cobalt-high speed steel by using polished rollers 2.0 mm in diameter. The remaining part of the rig was milled from a stainless steel block (A.I.S.I. type 316). The span was set at 13.0 mm. The tests were performed in a universal testing machine (Triax 50; Controls) with a crosshead speed of 1 mm/min. Specimens were tested at room temperature and humidity. The fracture load was recorded in N, and the flexural strength (σ) was calculated in MPa by using the following equation:

$$\sigma=\frac{3Pl}{2wb^2},$$

where P is the fracture load (N), l is the span (distance between the center of the supports) (mm), w is the width (mm), and b is the height (mm).

Data were tested to fit a normal distribution with the Kolmogorov-Smirnov test, and the homogeneity of variances was verified with the Levene test. The 1-way ANOVA was then performed (α=.05). The statistical analysis was performed with software (PASW Statistics 18; IBM). The Weibull characteristic strength (σ_0) and the Weibull modulus (m) were calculated according to ISO 6872:2008[38] from flexure strength data by rank order statistics with the following equation:

$$P_f=1-\exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right],$$

where P_f is the probability of failure between 0 and 1, σ is the flexural strength in MPa, σ_0 is the Weibull characteristic strength in MPa (the value at which 63.2% of the specimens fail), and m is the Weibull modulus.

RESULTS

Mean flexural strength, Weibull characteristic strength, and Weibull modulus are reported in Table 1. The Kolmogorov-Smirnov test confirmed the data distribution to be normal (P=.978, 2-tailed test). The 1-way ANOVA showed no statistically significant differences among the tested groups (P>.05). The power of the performed test with α=.05 was .907, higher than the .8 threshold.

The mean flexural strength recorded for the control group was 1235.7 ±86.9 MPa. The precolored group obtained a mean flexural strength of 1215.0 ±1117.5 MPa. Among the colored groups, the mean flexural strength varied between 1279.4 ±157.9 MPa (INF-N) and 1156.4 ±162.0 MPa (INF-M).

With regard to the Weibull modulus, the control group (noncolored) had a value of 16.8, and the precolored group had a value of 12.5. Among the colored groups, the highest values were calculated for INF-L (21.8) and the lowest for INF-M (8.4).

DISCUSSION

Because the results of the statistical analysis showed no statistically significant differences among noncolored, precolored, and infiltration-colored specimens, the coloring procedure did not significantly influence the flexural strength and Weibull characteristic strength of the tested materials. Hence, the research hypothesis was rejected.

The material investigated in the present study is based on pure zirconium oxides stabilized with yttria (Y-TZP). According to ISO 6872:2008,[38] this material must exhibit a mean flexural strength higher than 800 MPa to be classified as an ISO Class 6 ceramic (substructure ceramic for prostheses involving 4 or more units). The results obtained in the present study showed that even after a coloring procedure, the ISO Class 6 ceramic requirements were fulfilled.

To perform a coloring process, pigments (metal oxides) are added to Y-TZP. Huang et al[29] tested 5 types of oxides (Er_2O_3, CeO_2, Pr_6O_{11}, Fe_2O_3, and MnO_2) added in different combinations to raw zirconia powder to investigate their effect on the final color of zirconia. The authors showed that different oxides can be used to modify L*, a*, and b* color parameters even if no mechanical tests are performed after the color modification. In a similarly performed study, Yi et al[30] obtained colored zirconia with chromaticity properties comparable with those of the Vita In-Ceram YZ shade guide and concluded that the colored Y-TZP tested was suitable for clinical use. The efficacy of the coloring procedure from an esthetic viewpoint was reported also by Pecho et al,[31] who showed how colored and sintered zirconia specimens had smaller color differences compared to human dentin than noncolored zirconia specimens. Kaya[32] produced colored zirconia blocks by using different mixtures of 2 ready-to-press zirconia raw materials. The author used a different percentage of Fe_2O_3 content to color 3 mol% Y_2O_3 stabilized tetragonal zirconia to obtain the raw materials. After the full-sintering process, a change in the color of the specimens from white to yellowish brown was recorded and defined as suitable for dental color matching. No negative effects were found on either the tetragonal crystal phase formation or on the microhardness and fracture toughness values. Kaya[32] speculated that using zirconia raw materials with small amounts of Fe_2O_3, instead of adding Fe_2O_3 to the batches as a coloring agent, gave more suitable colors for dental restorations and was suitable for ceramic dental prostheses.

Table 1. Flexural strength (σ), Weibull characteristic strength (σ_0), and Weibull modulus (m) of tested materials

Description	σ Mean (MPa)	σ SD (MPa)	95% Confidence Interval for σ Mean (MPa)		σ_0 (MPa)	m
			Lower Bound	Upper Bound		
Noncolored (control)	1235.7	86.9	1175.0	1290.1	1275.0	16.8
Precolored	1215.0	117.5	1136.8	1293.5	1264.4	12.5
Infiltration-neutral	1279.4	157.9	1192.1	1369.3	1348.3	9.4
Infiltration-light	1219.8	68.0	1175.6	1262.3	1250.0	21.8
Infiltration-medium	1156.4	162.0	1055.8	1251.6	1224.7	8.4
Infiltration-intense	1248.5	102.4	1186.6	1310.3	1294.5	14.4

Statistical analysis was performed on flexural strength.

Coloring zirconia, however, has been reported to affect the mechanical properties of the material, even if a clear trend has not been identified in the literature. Ardlin[33] investigated the flexural strength of 2 shades of Denzir blocks, P0 (white) and P17 (yellowish), reporting a higher flexural strength with the P17 group. The author hypothesized that the higher strength of colored blocks might be related to components such as CeO_2, Fe_2O_3, and Bi_2O_3 added by the manufacturer to obtain the different shades. Pittayachawan et al[34] measured the biaxial flexural strength of colored and uncolored Y-TZP. No significant differences in biaxial flexural strength and Weibull modulus between uncolored and colored groups were reported, while a difference in microhardness was reported for some of the groups. Shah et al[22] showed that doping with metal oxides can modify color, density, grain size, and the total amount of open porosity of 3Y-TZP specimens. Biaxial flexural strength was reported to decrease when the concentration of solution increased, even if some low pH solutions (cerium acetate 1% and 5% and bismuth chloride 1%) did not affect the flexural strength. These findings were correlated with the type and concentration of dopant used.

Wen et al[35] showed that hue and brightness can be modified by adding Ce_2O and Er_2O_3 to raw zirconia powder, but a decrease of flexural strength (3PBT) of about 30% (1209 to 845 MPa) was found for colored specimens compared to noncolored specimens. Hjerppe et al[36] investigated the effect of green-stage color infiltration on biaxial strength and surface microhardness of ICE Zirkon blocks. They reported a significant reduction in strength compared to the control group when shading was performed. A prolonged shading time further decreased the strength. Vickers surface microhardness values tended to decrease, even if the differences were not statistically significant. No changes from the tetragonal to monoclinic phase in the zirconia specimens were found after color shading.

The importance of shading time was also reported by Liu et al,[37] who found that increasing the shading time produced a lower biaxial flexural strength. The effects of doping zirconia with cerium and erbium oxides on the final color were reported by Li et al.[23] The mixture of Ce_2O and zirconia produced a pale yellow color, and the mixture of Er_2O_3 and zirconia produced a pale pink color. An orange color can be obtained by mixing the oxides. The color intensified with dose increase. The study reported that the flexural strength of the colored specimens decreased with the increase of pigment content. In particular, a reduction in the flexural strength of the materials with added cerium dioxide or erbium oxide alone was reported. However, cerium dioxide led only to a small decrease in flexural strength, while erbium oxide significantly reduced flexural strength. The flexural strength of the specimens with the added mixture of cerium dioxide and erbium oxide decreased with the increase of cerium dioxide and erbium oxide used.

Some oxides, such as Fe_2O_3, CeO_2, Er_2O_3, Pr_6O_{11}, Bi_2O_3, and MnO_2, can modify the color of Y-TZP. However, depending on the concentration, the mechanical characteristics of the materials can be modified. CeO_2 and Er_2O_3 at low concentration seem able to change the color of zirconia without reducing its flexural strength.

The results obtained in the present study showed that the dopants used by the manufacturer to modify the shade of the zirconia, both in precolored blocks and in coloring liquids, did not affect the flexural strength. The green-stage color infiltration was performed strictly following the time indicated by the manufacturer, avoiding a possible reduction of flexural strength caused by prolonged immersion.[36] A limitation of this study is that the chemical composition of the coloring liquid was not investigated. The 3PBT selected for the present study is considered a valid method for comparing the mechanical behavior of brittle materials[39] and is reported in ISO 6872:2008 as a method of assessing the flexural strength of CAD/CAM ceramic materials.[38] Nevertheless, it is not entirely representative of the actual clinical situation, and clinical studies should be performed to assess the behavior of shaded zirconia under clinical conditions, particularly under long-term use.

CONCLUSIONS

Within the limits of the present study, it can be concluded that factory preshading or the use of Vita In-Ceram YZ coloring liquids according to the manufacturer's instructions does not negatively affect the flexural strength of Vita In-Ceram YZ for the CEREC CAD/CAM system.

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