

Effect of Coloring–by-Dipping on Microtensile Bond Strength of Zirconia to Resin Cement

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Abstract

Objectives: Studies on the effect of coloring procedures on the bond strength of zirconia to resin cement are lacking in the literature. This study evaluated the effect of dipping of zirconia ceramic in different liquid color shades on the microtensile bond strength (MTBS) of zirconia ceramic to resin cement.

Materials and Methods: This in vitro study was conducted on 100 microbar specimens divided into five groups of B2, C1, D4, A3 and control (not colored). To prepare the microbars, 20 white zirconia ceramic blocks, measuring 5×11×11 mm, were dipped in A3, B2, C1 or D4 liquid color shades for 10 seconds (five blocks for each color shade) and five blocks were not colored as controls. All the zirconia blocks were sintered in a sintering furnace. Composite blocks of similar dimensions were fabricated and bonded to zirconia ceramic blocks using Panavia F 2.0 resin cement. Zirconia-cement-composite blocks were sectioned into microbars measuring 1×1×10 mm. The MTBS of microbars was measured by a testing machine. Data were analyzed using one-way ANOVA and Tukey's test. All tests were carried out at 0.05 level of significance.

Results: Statistically significant differences were found among the groups in MTBS ($P < 0.001$). The D4 group had the highest MTBS value (39.16 ± 6.52 MPa).

Conclusion: Dipping affected the MTBS of zirconia ceramic to Panavia F 2.0 resin cement; however, a similar pattern of change was not seen due to the different liquid color shades.

Keywords: Yttria stabilized tetragonal zirconia; Resin Cements; Prosthesis coloring

Journal of Dentistry, Tehran University of Medical Sciences, Tehran, Iran (2015; Vol. 12, No. 6)

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Received: 28 August 2014

Accepted: 27 March 2015

INTRODUCTION

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic has extensive applications in restorative dentistry due to excellent mechanical properties and biocompatibility [1,2]. Zirconia computer-aided design and computer-aided manufacturing (CAD/CAM) systems have enabled the fabrication of Y-TZP dental

ceramic restorations. Use of resin cements between the tooth structure and the restorative material increases retention especially in cases with short crown height [3,4]. Additionally, application of adhesive bonding improves marginal seal and prevents secondary caries [5-8].

The MTBS of zirconia ceramic to cement has been evaluated by several researchers [9,10].

The MTBS is influenced by the choice of cement [11,12], surface treatment of zirconia ceramic [9,12-18], bonding technique [19-21], water storage [22,23] and thermocycling [24-26]. Among various resin cements, Panavia F 2.0 has been recommended to achieve the highest MTBS of zirconia ceramic to cement [11,26-28].

As Y-TZP ceramic is white in color, it can affect the optical characteristics of the restoration. Thus, by coloring the Y-TZP ceramic, more favorable esthetic results can be obtained [29].

Various techniques have been developed for coloring of zirconia CAD/CAM systems such as adding metallic pigments to the zirconia powder by the manufacturers, dipping the zirconia framework in color liquids before sintering and using different liner materials after sintering the zirconia framework. In dipping method, the milled zirconia ceramic framework is immersed in a color liquid before sintering for a predetermined time by a dental technician [30,31]. Liquid color shades, marketed by the manufactures, can infiltrate into the zirconia surface structure and color the zirconia. Mechanical properties of zirconia can be influenced by dipping in liquid color shades. Hjerpe et al. [31] showed that liquid color shades affected biaxial flexural strength and surface microhardness of zirconia. Shah et al. [32] found that the flexural strength of zirconia decreased as the concentration of the liquid color shades increased; however, resistance to low temperature degradation was not affected by the coloring process.

Studies on the influence of coloring by dipping on the bond strength of zirconia ceramic to resin cement are lacking in the literature. Therefore, the aim of the current in vitro study was to evaluate the effect of coloring by dipping on the bond strength of zirconia ceramic to resin cement. The null hypothesis of this study was that the coloring by dipping in different liquid color shades

would not affect the MTBS of Y-TZP dental ceramic to Panavia F 2.0 resin cement.

MATERIALS AND METHODS

This in vitro study was conducted on 100 microbar specimens divided into five groups of B2, C1, D4, A3 and control (not colored). Twenty microbar specimens were prepared for each group. Each microbar specimen was then tested to measure the MTBS of zirconia ceramic to resin cement. This process was done precisely as follows:

Specimen preparation:

Initially, 25 zirconia blocks measuring 5×11×11 mm were milled from Y-TZP dental ceramic blanks (Ice Zircon Translucent, Zirkon Zahn, Bruneck, Italy) using a CAD/CAM system (Zirkon Zahn CAD/CAM System 5-TEC, Zirkon Zahn, Bruneck, Italy) with CAD/CAM milling burs (CAD/CAM Milling Bur 1L & 2L, Zirkon Zahn, Bruneck, Italy). These blocks were divided into five groups of five blocks each. The blocks of each group were dipped in a liquid color shade including B2, C1, D4 and A3 (Color Liquid Water-based, Zirkon Zahn, Bruneck, Italy) for 10 seconds (recommended by the manufacturer) and one group was not colored as the control.

The colored blocks were then dried using a drying lamp (Zirkon lamp 250, Zirkon Zahn, Bruneck, Italy) for 45 minutes as recommended by the manufacturer. All the zirconia blocks were then sintered at 1,480°C for eight hours in the sintering furnace (Zirkonofen 700, Zirkon Zahn, Bruneck, Italy) according to the instruction recommended by the manufacturer.

In order to prepare composite blocks as bases for the zirconia blocks with similar dimensions, an impression was taken of each zirconia block using condensation silicone putty material (Speedex, Coltene, Altstätten, Switzerland).



Fig. 1. The microbar specimen under a microscope at $\times 25$ magnification



Fig. 2. The fractured microbar specimen after MTBS testing

A light-polymerized composite resin (Z100 Restorative, 3M ESPE, St. Paul, MN, USA) was applied in 2 mm layers to the molds made of impression material and was polymerized by a light-curing unit (Elipar FreeLight 2, 3M ESPE, St. Paul, MN, USA) from both sides for 60 seconds with an intensity of 800 mW/cm^2 .

The composite blocks were made as such, polished with 800 grit silicon carbide abrasive papers for 10 minutes and cleaned in an ultrasonic bath (Elmasonic S-30, Dentec, North Shore, Australia) containing 98% ethanol for 10 minutes. Afterwards, the surfaces of zirconia blocks were sandblasted with 110μ aluminum oxide particles at 0.3 MPa pressure for 15 seconds using a sandblaster (Korostar Plus, Bego, Bremen, Germany) held at a distance of 10 mm and perpendicular to the block surface. The blocks were rinsed with water for 20 seconds and air dried for five seconds using air spray. The resin cement (Panavia F 2.0, Kuraray, Tokyo, Japan) was prepared as instructed by the manufacturer. The zirconia blocks were then bonded to the composite blocks using resin cement under a 50 N compressive force in order for the excess cement to leak out. An oxygen inhibiting gel (Oxyguard II, Kuraray, Tokyo, Japan) was applied to the external surface of the cement. After five minutes, the blocks were light cured from four different directions for 40 seconds with the same light-curing unit, rinsed to remove the gel and stored in distilled water at 37°C for 24 hours.

To prepare specimens for testing, zirconia-cement-composite blocks were fixed in a metal mold. The mold was placed in a low-speed cutting machine (Mecatome, T210A, Presi, Grenoble, France). The blocks were sectioned with a diamond saw under water cooling.

The first section with an approximate thickness of one millimeter was discarded because the presence of excess cement or lack of cement in this section could confound the results. Two series of sections were vertically cut with a rotational speed of 600 rpm and forward motion speed of 6 mm/min to prepare microbars measuring $1 \times 1 \times 10$ mm from each zirconia-cement-composite block. The microbars were evaluated under a stereomicroscope (SMZ 800, Nikon, Sendai, Japan) at $\times 25$ magnification (Fig.1). In case of any defect, the defective specimen was excluded from the study. The microbar specimens were stored in distilled water at 37°C for 72 hours. Twenty microbar specimens were prepared for each group (B2, C1, D4, A3 and control).

Testing:

The microbar specimens were glued to the jig of the microtensile tester (Microtensile Tester, Bisco, Schaumburg, IL) using cyanoacrylate glue (Loctite, Henkel, Dusseldorf, Germany) and fixed. The microbar was positioned parallel to the long axis of the machine to minimize bending stresses.

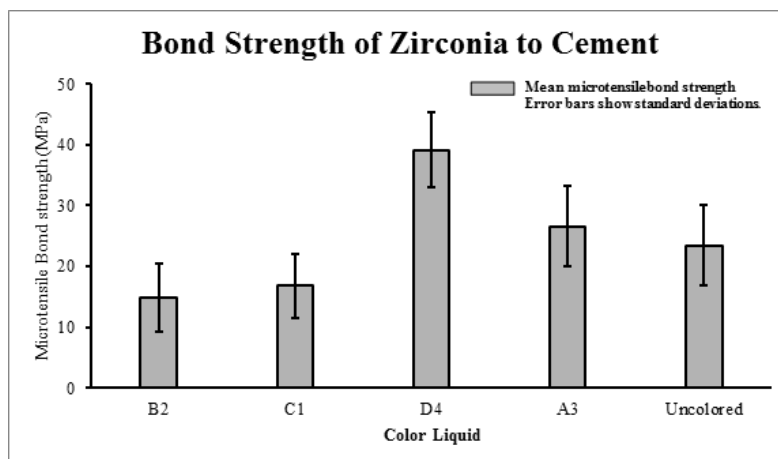


Fig. 3. The mean and standard deviation of MTBS for the five groups

The crosshead speed of the machine was adjusted at 1 mm/min. The testing machine applied force until the microbar fracture occurred (Fig. 2) and measured the microtensile force at fracture in Newton. The fractured surfaces were evaluated under a stereomicroscope at $\times 40$ magnification. In case of any fracture in the composite or between the composite and cement, the specimen was excluded from the study. The bonding surface area was measured by a digital caliper adapted to the machine. The MTBS was calculated using this formula: $R=F/A$, where F is the failure force in N, A is the interface area in mm^2 and R is the MTBS of zirconia to cement in MPa. The MTBS values were analyzed for the five groups using one-way ANOVA. Pairwise comparison of groups was carried out using Tukey's Post Hoc test.

All tests were carried out at 0.05 level of significance. Statistical tests were done using SPSS 20 (SPSS Inc., Chicago, IL, USA).

RESULTS

The mean and standard deviation of MTBS of microbar specimens in the five groups of B2, C1, D4, A3 and control were 14.77 ± 5.59 MPa, 16.86 ± 5.26 MPa, 39.16 ± 6.52 MPa, 26.61 ± 6.59 MPa, and 23.44 ± 6.56 MPa, respectively (Fig. 3) (Table 1). One-way ANOVA showed statistically significant differences in the MTBS values of the five groups ($P < 0.001$). The results of Tukey's pairwise comparison revealed statistically significant differences in the MTBS values between B2 and D4 ($P < 0.001$), B2 and control ($P < 0.001$), B2 and A3 ($P < 0.001$), C1 and D4 ($P < 0.001$), C1 and A3 ($P < 0.001$), C1 and control ($P < 0.009$), D4 and A3 ($P < 0.001$) and

Table 1. Central dispersion measures for MTBS values of microbar specimens in the five groups of B2, C1, D4, A3 and control

Color shade	Mean (MPa)	SD	Standard error	Minimum	Maximum	Range
B2	14.77	5.59	1.25	12.16	17.38	6.3-25.3
C1	16.86	5.26	1.18	14.39	19.32	10.3-28.8
D4	39.16	6.52	1.46	36.11	42.21	28.5-49.2
A3	26.61	6.59	1.47	23.52	29.69	15.0-38.4
Uncolored	23.44	6.56	1.47	20.37	26.51	10.7-32.0

D4 and control ($P < 0.001$); whereas, the differences between B2 and C1 ($P = 0.82$), and A3 and control ($P = 0.48$) were not statistically significant.

DISCUSSION

The present study evaluated the MTBS of the prepared microbar specimens in four groups dipped in different liquid color shades and one control group. A similar pattern of change in the MTBS was not seen after dipping in different liquid color shades. In comparison with the control group, B2 and C1 liquid color shades decreased the MTBS; while A3 liquid color shade did not decrease the MTBS and D4 liquid color shade increased the MTBS. In Zirkozahn zirconia system, B2 and C1 liquid color shades had negative effects, D4 liquid color shade had positive effect and A3 liquid color shade had no effect on the MTBS of zirconia to Panavia F 2.0 resin cement. Hence, the null hypothesis of the study was refuted.

Hjerppe et al. [31] revealed that biaxial flexural strength and surface microhardness of zirconia discs were influenced by the color liquids and the zirconia colored by D4 liquid color shade had the highest MTBS compared to A3, B1, C4 and D2. The D4 liquid color shade in contrast to the other tested color liquids did not cause a statistically significant reduction in MTBS [31]. Although the current study evaluated the MTBS of colored zirconia to cement, both investigations showed changes in zirconia properties following dipping for coloring.

According to the result of a study by Hjerppe et al, [31] all the tested color liquids except for the D4 shade had negative effects on the biaxial flexural strength of zirconia. A similar result was obtained in the current study showing the positive effect of D4 liquid color shade on MTBS of zirconia to cement. Both investigations demonstrated the negative effects of B and C liquid color shades. The changes in the zirconia properties were reported in both studies.

Shah et al. [32] performed an in vitro study to investigate the effect of coloring by dipping in cerium acetate, cerium chloride, or bismuth chloride with three different concentrations (1, 5 and 10%) on the microstructure, color, flexural strength and aging resistance of tetragonal zirconia for dental applications. The results showed the effects of liquid color shades on zirconia including slight increase in lattice parameters of tetragonal zirconia, increase of grain size and decrease of flexural strength with an increase in concentration of both cerium salts. However, the liquid color shades did not affect the density or the resistance to low temperature degradation. A decrease in flexural strength at higher concentrations was attributed to an increase in open porosity [32]. Although our study evaluated the MTBS of colored zirconia to cement and used different liquid color shades compared to the study by Shah et al, [32] both investigations showed changes in zirconia properties following coloring by dipping. Evidence suggests that the bonding mechanism of zirconia to resin cement is mainly related to the bond between the metal oxides in Y-TZP ceramic and 10-hydroxydecyl dihydrogen phosphate (MDP) in a MDP-containing resin cement such as Panavia F 2.0 [26,27,33-35]. Vinyl group of MDP reacts with monomers in resin cement when resin is polymerized; while phosphate group of MDP bonds strongly to metal oxides of Y-TZP ceramic. These oxides are added to zirconia during the manufacturing and coloring processes [36]. It seems that some metal oxides at the zirconia surface microstructure play a significant role in the bond. Meanwhile, the bond may be influenced by dipping because liquid color shades infiltrate into the zirconia surface. Therefore, effects of coloring by dipping on the bond strength of zirconia to cement may be explained by two factors: the liquid color shade composition and its effect on zirconia surface microstructure.

The composition of the liquid color shade may impact the bond strength of zirconia to cement in two ways namely by the type of added metal oxides to zirconia structure and tetragonal/monoclinic phase transformation. The metal oxides added to Y-TZP ceramics during the ceramic processing, manufacturing or coloring can influence the zirconia properties. It has been well confirmed that silica or alumina directly influences grain morphology, microstructure and stability of zirconia [37,38]. Ceria acts as a sintering aid [39], increases the grain size [39] and improves aging resistance [40] of Y-TZP. Bismuth acts as a stabilizer and a sintering aid for tetragonal zirconia [41]. Addition of CaO, MgO, CeO and Y₂O₃ to pure zirconia allows generating multi-phase materials [42]. MnO₂ is associated with rounded pore formation in Y-TZP [43]. CuO suppresses the transformation of tetragonal to monoclinic phase [44]. Pittayachawn et al. [45] evaluated the biaxial flexural strength, hardness and fatigue life of colored and uncolored zirconia in the Lava system. Although the colored zirconia used in their study was prefabricated by the manufacturer, the results showed a difference in oxygen content, which was higher in colored zirconia. This was attributed to the presence of oxides. No difference was reported in biaxial flexural strength between colored and uncolored zirconia with the exception of two shades; while there was a significant difference in hardness among the groups. Additionally, the uncolored and colored specimens showed some differences in the grain size and microstructure. The uncolored samples revealed the spectra as expected for tetragonal and cubic zirconia, while the colored samples were very different [45]. Ardlin [36] determined chemical stability and effect of aging (4% acetic acid at 80°C for 168 hours) on flexural strength, surface and crystalline structure of colored and uncolored zirconia in the Denzir system and in contrast to Pittayachawn et al, [45] he found that the

flexural strength of yellow colored zirconia was higher than that of uncolored zirconia. Ardlin [36] suggested that it might be related to the components such as CeO₂, Fe₂O₃ and Bi₂O₃ added during the manufacturing process to obtain different shades. This difference in results may be caused by the type of oxides added by the different manufacturers to the Lava system (a dental Y-TZP ceramic) used by Pittayachawan et al, [45] versus the Denzir system (a dental Mg-TZP ceramic) used by Ardlin [36]. In both studies, the effect of coloring by the manufacturer on the flexural strength of zirconia was evaluated; while the current study evaluated the effect of coloring by dipping on the bond strength. Such controversy in results may be related to the coloring technique and the type of oxides added. Accordingly, in coloring by dipping, where liquid color shades containing metal oxides are utilized before sintering, some metal oxides may infiltrate into zirconia surface microstructure and may be added to or replace the existing ones. This phenomenon may affect the bond strength of zirconia to Panavia F 2.0. This effect, based on the nature of the oxides, may be positive, negative or neutral. It may be the reason for positive effect of D4 liquid color shade, negative effect of B2 and C1 liquid color shades and neutral effect of A3 liquid color shade on the MTBS of zirconia to Panavia F2.0 in the current study. The composition of the liquid color shade may be responsible for the weakening effect of A3, B1 and C4 liquid color shades on the zirconia hardness showed by Hjerpe et al, [31] the weakening effect of B2 and C1 liquid color shades and the improving effect of D4 liquid color shade on the MTBS of zirconia to cement (showed by the current study). The specific effect of liquid color shades on zirconia surface microstructure may be another important factor explaining the effect of coloring on zirconia bond to cement. The concentration of yttrium, which is the primary stabilizing element of zirconia used in dental

restorations, was found to be higher at grain boundaries and surfaces compared with the grain interior [21]. Yttrium also contributes to other dynamic properties such as grain sliding, rearrangement movements and plastic deformation [46,47]. There may be a balance between metal oxides and yttria in Y-TZP, which protects the tetragonal structure. Coloring by the manufacturer can affect this structure [45]. Infiltration of the metal oxides during dipping may promote or suppress this balance and may create a shift in tetragonal/monoclinic phase transformation. This shift may alter the surface microstructure. Moreover, the zirconia surface structure may be influenced by the dipping time and the concentration of color liquids. These factors may potentially affect the infiltration of oxides into the zirconia surface. Hjerpe et al. [31] found that prolonging the dipping time from 3 to 60 seconds lowered the zirconia biaxial flexural strength. Dipping time had no effect on zirconia microhardness, but there were some differences among different shades [31]. The effect of concentration of color liquids on zirconia surface and strength was shown by Shah et al [32]. They demonstrated that solutions with higher concentrations had a greater negative effect on the zirconia strength. Consequently in coloring by dipping, the interaction effect of all the above-mentioned factors may influence the surface composition and surface structure of zirconia, which can directly influence the bonding mechanism of zirconia to monomer-based phosphate cements such as Panavia F 2.0. The current study results can be interpreted from the perspectives of the manufactures, dental clinicians and investigators. The manufacturers should modify the color liquids with poor performance to prevent their negative effects on the bond strength of zirconia to resin cement. The clinicians should use the liquid color shades (B2 and C1) with poor performance with caution specially in cases with decreased clinical crown height,

decreased bonded surface area, compromised retention of restoration or heavy functional and parafunctional occlusal loads.

The investigators can take advantage of the liquid color shades (D4) with optimal performance to improve the zirconia surface bond. Further investigations are required in this regard [18,21]. The results of our study revealed the effects of coloring by dipping on MTBS of zirconia to cement. Another study is recommended using the thermocycling process to better simulate the clinical conditions.

Using only the Zirkozahn system, specific liquid color shades and 10 seconds of dipping time were the limitations of the current study. Therefore, more studies on the mode of failure, elemental composition of Y-TZP, dipping time, different shades and different zirconia systems are recommended.

CONCLUSION

Within the limitations of this study, it was concluded that coloring by dipping affected the MTBS of zirconia ceramic to Panavia F 2.0 resin cement; however, a similar pattern of change in the MTBS was not seen among the different liquid color shades.

ACKNOWLEDGMENTS

The authors would like to thank the Research Deputy of Dental School of Shahid Beheshti University of Medical Sciences for financially supporting this project (grant# 324).

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