Dipping Impact on the Bond Strength between Zirconia Ceramic and a Resin Cement

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Introduction: Dipping technique is commonly used by dental technicians to color zirconia ceramics; however, its impact on the bond between zirconia and resin cements is unclear. This in vitro study aimed to evaluate the microtensile bond strength (μTBS) between a zirconia ceramic and Panavia F2.0 resin cement after dipping in coloring liquids with different shades. **Materials and Methods**: Twenty zirconia blocks were dipped in four different shades of coloring liquids including A3, B2, C1, and D4, while five blocks were not colored for the control. All zirconia blocks were cemented to corresponding composite blocks using Panavia F2.0 resin cement. Zirconia-cement-composite blocks were cut into one hundred microbar specimens (1×1×10 mm). The specimens were divided into five studied groups according to their shade (A3, B2, C1, D4, control) and were subjected to an aging process. A microtensile tester applied tensile forces to the specimens, till the fracture occurred, and measured the μTBS values. One-way ANOVA and Tukey's Post Hoc were used to analyze the data ($P<0.05$). Optical microscopy and scanning electron microscopy were employed to evaluate failure modes and surface structure. **Results:** The μTBS values for the groups showed significant differences ($P<0.0001$). D4 had the highest, and B2 had the lowest μTBS values. C1 ($P=0.69$) and A3 ($P=0.89$) showed no significant differences in the μTBS values compared with the control. Failure modes evaluation represented the lowest rate of adhesive failure for D4 (10%), and the highest in this respect for B2 (80%). **Conclusion:** Depending on the shade of coloring liquids, dipping had positive, negative, or no effects on the μTBS of zirconia ceramic to Panavia F2.0 resin cement.

Keywords: Prosthesis coloring; Resin cements; Zirconia

Introduction

Zirconia ceramics have represented acceptable mechanical properties and appropriate biocompatibility (1, 2). In the recent decade computer-aided design and computer-aided manufacturing (CAD/CAM) systems have been well established in dentistry to fabricate zirconia ceramic restorations. Besides, resin cements have been improved to enhance retention and marginal seal between the tooth structure and zirconia restorations (3-8).

In order to evaluate the bond of zirconia ceramic to resin cement, the microtensile bond strength (μTBS) has been measured (9, 10) .The literature has reported that the μTBS can be influenced by type of cement (11, 12), surface treatment of ceramics (9, 12-18), bonding method (19-21), water aging (22, 23), thermocycling (24-26) , coloring (27), and particle deposition (28). Panavia F2.0 resin cement has represented the strongest bond to zirconia ceramic among different resin cements because of its chemical composition (11, 26, 27, 29, 30).

Three techniques have been employed to color zirconia restorations including: coloring zirconia powder to produce pre-colored zirconia ceramics, coloring non-sintered zirconia through dipping in coloring liquids, and lining sintered zirconia by colorant materials (27). Ardlin (31), Pittayachawn *et al.* (32), and Sedda *et al.* (33) in separate studies reported the effects of pre-colored zirconia on zirconia properties such as flexural strength, hardness and fatigue life; however, their findings were controversial. Aboushelib *et al.* (34) advocated that liners weakened the bond between zirconia core and veneering porcelain. Dipping technique for zirconia has been proposed by Vichi *et al.* (35) as an alternative to the use of liners. It seems that among different coloring techniques, the dipping is the most commonly used technique which is performed by dental technicians. The effects of this laboratory procedure on zirconia properties have been concerned by investigators. Hjerppe *et al.* (36) indicated that the dipping technique influenced the biaxial flexural strength of zirconia. Shah *et al.* (37) revealed that the flexural strength of zirconia ceramics decreased as the result of an increase in the concentration of coloring liquids. Mahshid *et al.* (27) reported that the coloring by dipping affected the bond of zirconia ceramic to resin cement; however, a similar pattern of change in the bond strength was not observed among tested coloring liquids.

Regarding the importance of the bond of zirconia ceramic to resin cement especially in cases with the compromised retention of restoration, dipping impact on this bond should be clearly understood. Therefore, the aim of this in vitro study was to evaluate the µTBS between a zirconia ceramic and Panavia F2.0 resin cement after dipping in coloring liquids with different shades. The null hypothesis of the study was that the dipping would not affect the µTBS between zirconia ceramic and Panavia F2.0 resin cement.

Materials and Methods

One hundred microbar specimens (n=100) were prepared and divided into five groups of A3, B2, C1, D4, and control (each group of twenty). The sample size was determined regarding $\alpha=0.05$ and $\beta=0.1$. All microbar specimens were subjected to an aging process and then tested to measure the µTBS of zirconia ceramic to resin cement.

A CAD/CAM system (Zirkonzahn CAD/CAM System 5-TEC, Zirkonzahn, Bruneck, Italy) milled zirconia ceramic blanks (Ice Zircon Translucent, Zirkonzahn, Bruneck, Italy) to fabricate twenty five zirconia blocks (5×11×11 mm). These blocks were divided into five groups according to the shade of coloring liquids used (Color Liquid Water-based, Zirkonzahn, Bruneck, Italy). The four shade groups included A3, B2, C1, and D4, while one group was not colored for the control. The blocks were dipped in coloring liquids for 10 s. The dipped blocks were dried by a lamp (Zirkonlamp 250, Zirkonzahn, Bruneck, Italy) for 45 minutes. All the blocks sintered at 1500°C for eight hours in a sintering furnace (Zirkonofen 700, Zirkonzahn, Bruneck, Italy). The whole procedure was performed according to the instruction recommended by the manufacturer.

Composite blocks were prepared to cement to the zirconia blocks. For this purpose, a putty silicone impression (Speedex, Coltene, Altstatten, Switzerland) was taken of each zirconia block. A light-polymerized composite resin (Z100 Restorative, 3M ESPE, St. Paul, MN, USA) was placed in layers in the putty molds. A light-polymerizing unit (Elipar FreeLight 2, 3M ESPE, St. Paul, MN, USA) was applied to polymerize the composite resin incrementally for 60 s with an intensity of 800 mW/cm^2. The composite blocks were polished with 800 grit silicon carbide abrasive papers for 15 minutes and cleaned in an ultrasonic bath containing 98% ethanol for 15 minutes. The surfaces of zirconia blocks were subjected to a sandblaster (Korostar Plus, Bego, Bremen, Germany) with 110µ aluminum oxide particles at 0.3 MPa pressure for 15 s. The sandblaster was set perpendicular to the blocks at a distance of 10 mm. The blocks were cleaned with water for 20 s and dried with compressed air. The zirconia blocks were then cemented to the composite blocks by use of Panavia F2.0 resin cement (Panavia F 2.0, Kuraray, Tokyo, Japan), according to the manufacturer's instruction, under a 50 N compressive force for 5 minutes. An oxygen inhibiting gel was employed to cover the external surface of the cement during polymerization. Then the same light-polymerizing unit was used to polymerize the cement from four different directions for 60 s. The gel was rinsed and removed. The zirconia-cement-composite blocks were stored in distilled water at 37°C for 24 hours.

A low speed cutting machine (Mecatome, T210A, Presi, Grenoble, France) cut the zirconia-cement-composite blocks with a rotational speed of 600 rpm and forward motion speed of 6 mm/min to prepare microbar specimens measuring 1×1×10 mm. The first external cut was eliminated because of the possibility of cement insufficiency. The specimens were observed under a stereomicroscope (SMZ 800, Nikon, Sendai, Japan) at ×25 magnification to detect defective ones and to discard them from the study. The specimens were stored in distilled water at 37°C for 72 hours and were then subjected to thermocycling for 10,000 cycles at 5°C-55°C with 20 s in water bath and 10 s of dwell time.

The specimens were fixed to the jig of a microtensile testing machine (Microtensile Tester, Bisco, Schaumburg, IL), while placed parallel to the long axis of the machine for applying correct force direction. The crosshead speed was adjusted at 1 mm/min. The machine applied tensile forces till the specimen fractured. The failure force was recorded for each specimen. The fractured surfaces were observed by the same stereomicroscope at 40 × magnification. In case of any fracture in the composite or interface of the composite and cement, the specimen was discarded from the study. The bonding area was measured by a digital caliper. The µTBS was determined by this formula: R=F/A (F: failure force in N, A: bonding area in mm^2, R: µTBS in MPa).

The fractured specimens were evaluated under the same stereomicroscope at 40 × magnification to determine modes of failure. In adhesive failures, the fracture was observed at the interface of cement and zirconia. In cohesive failures, the fracture occurred within the cement while cement coating existed on both zirconia and composite surfaces. Mixed failures were a combination of adhesive and cohesive failures. Additionally, two specimens of each group were prepared for scanning electron microscopy (SEM) analysis at 5000 × magnification in order to observe the zirconia surface structure after fracture. A software (SPSS 21, SPSS Inc., Chicago, IL, USA) was employed to analyze the data. One-way ANOVA was used to analyze the µTBS values among the five groups. Pairwise comparisons of the groups were performed by use of Tukey's Post Hoc. $P<0.05$ was considered for all the tests.

Results

The means and standard deviations of µTBS values of microbar specimens in the five groups of B2, C1, D4, A3 and control were 9.34±1.63, 14.21±6.71, 23.54±4.12, 14.83±4.72, and 16.37±7.08

Table 1. Measures of central dispersion for the μTBS values of microbar specimens in studied groups

Group	Mean	Standard Deviation	Standard error	Minimum	Maximum	Range
B2	9.34	1.63	0.37	8.58	10.1	6.3-12.5
C1	14.21	6.71	1.5	11.07	17.34	9.4-33.2
D4	23.54	4.12	0.92	21.6	25.46	18.2-32.3
A3	14.83	4.72	1.06	12.61	17.04	9.3-22.6
Control	16.37	7.08	1.58	13.05	19.68	7.9-34.8

Table 2. Modes of failure of microbar specimens in studied groups

Group	Adhesive (number, percentage)	Mixed (number, percentage)
B2	16 , 80 %	4 , 20 %
C1	13 , 65 %	7 , 35 %
D4	2 , 10 %	18 , 90 %
A3	7 , 35 %	13 , 65 %
Control	9 , 45 %	11 , 55 %

MPa, respectively (Table 1)(Figure 1). One-way ANOVA showed statistically significant differences in the μTBS values of the studied groups (*P*<0.0001). Tukey's pairwise comparisons showed significant differences in the μTBS values between B2 and D4 (*P*<0.0001), B2 and control (*P*<0.0001), B2 and C1 (*P*<0.033), B2 and A3 (P<0.011), D4 and control (*P*<0.0001), D4 and C1 (*P*<0.0001) and D4 and A3 (*P*<0.0001). However, no significant differences were found in this respect between C1 and control (*P*=0.69), A3 and control (*P*=0.89), and C1 and A3 (*P*=0.99). The general ranking of the μTBS for the examined groups based on the statistical analysis was D4 > control ≥ A3 ≥ C1 > B2.

Evaluation of the modes of failure showed adhesive failure rates of 80%, 65%, 10%, 35%, and 45% for the groups of B2, C1, D4, A3, and control, respectively (Table 2). Also the rates of mixed failure for the groups of B2, C1, D4, A3, and control were 20%, 35%, 90%, 65%, and 55%, respectively (Table 2). No cohesive failure was detected.

Discussion

The current research assessed the μTBS of microbar specimens in four groups dipped in different coloring liquids and one control group. All specimens were subjected to a similar aging procedure including water aging and thermocycling. According to the results (Table 1 and Figure 1) different changes in the μTBS values were observed after dipping. In comparison with the control, the coloring liquid B2 decreased the μTBS significantly, while coloring liquids A3 and C1 did not decrease the μTBS significantly and the coloring liquid D4 significantly increased the μTBS. Consequently in Zirkonzahn zirconia system, the coloring liquid B2 had negative effect, coloring liquid D4 had positive effect and the coloring liquids A3 and C1 had no effect on the μTBS of zirconia to Panavia F 2.0 resin cement. Thus the null hypothesis of the study was partially rejected.

Evaluation of the failure modes of specimens revealed that the group D4 had the highest rate in mixed failures; followed by the groups A3, control, C1, and B2, respectively (D4>A3>Control>C1>B2). On the contrary, the group D4 had the lowest rate in adhesive failures; followed by the same mentioned arrangement (D4<A3<Control<C1<B2) (Table 2). According to the definition of failures, the adhesive failure expresses a weak bond between cement and zirconia. Comparing the results in Table 1 and Table 2 shows the group D4 with the highest μTBS value and the lowest rate of adhesive failure. Conversely, the group B2 displays the lowest μTBS value and the highest rate of adhesive failure. This harmony in the results demonstrated in Table 1 and Table 2, seems to be rational. Furthermore, SEM analysis displayed a more irregular and uneven surface for the specimen D4 than the other specimens (Figure 2). These observations explain the reason for the highest μTBS value of the group D4 among the studied groups.

Mahshid *et al.* (27) performed an in vitro study to evaluate the effect of coloring by dipping on the μTBS of zirconia to a resin cement and revealed that the coloring liquid D4 increased the μTBS, the coloring liquids C1 and B2 decreased the μTBS, and the coloring liquid A3 had no effect on the μTBS. The current study assessed the dipping effect on the μTBS under aging conditions which seems to be a better simulation of clinical conditions. This may be a reason for the results dissimilarities. Moreover, the present study surveyed the mode of failure of the specimens which was not evaluated by Mahshid *et al.* (27).

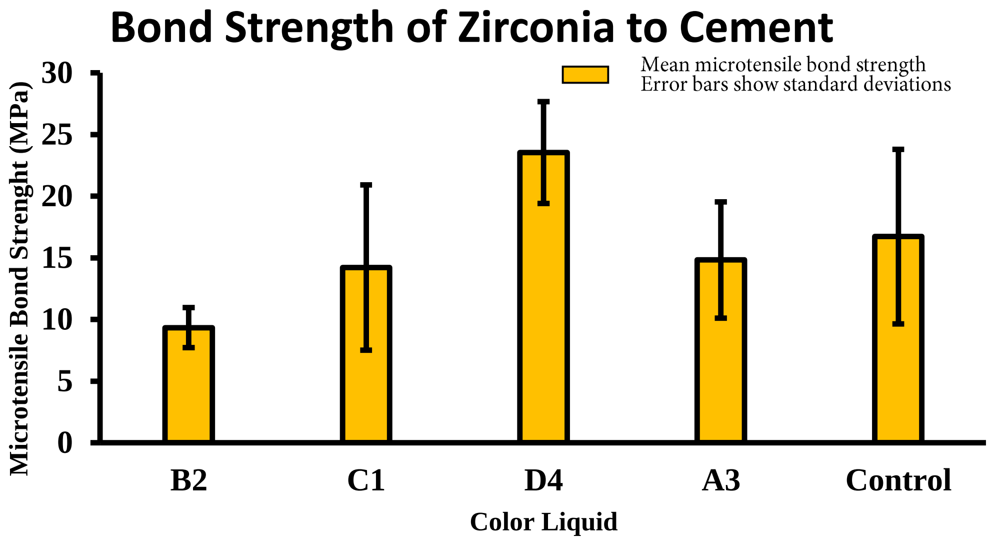

Figure 1. The means and standard deviations of μTBS for studied groups

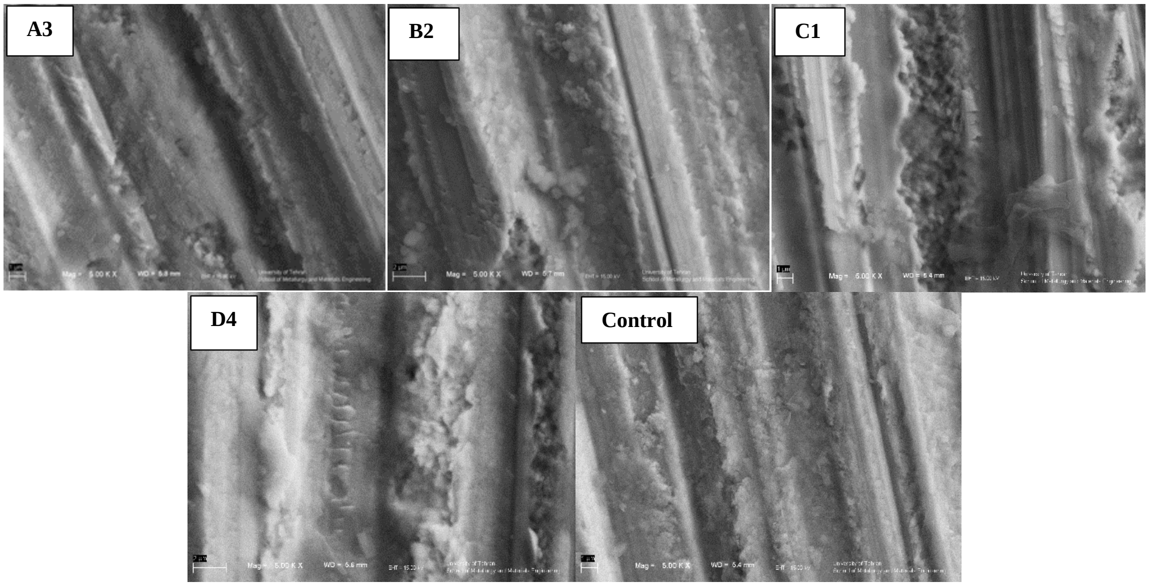

Figure 2. Representative SEM specimens of zirconia surfaces in studied groups (magnification 5000)

Shah *et al.* (37) studied the influence of different coloring liquids with different concentrations on the microstructure, color, flexural strength, and aging resistance of zirconia ceramic. The specimens were analyzed after aging in autoclave for 10 hours. Their findings revealed the impacts of coloring liquids on zirconia including zirconia lattice parameters growth, grain size enlargement and flexural strength reduction caused by an increase in concentration of cerium salts, while coloring liquids did not impact the density and the resistance to low temperature degradation (37). Though the current study estimated the bond of zirconia to cement and tested different coloring liquids and aging process compared to the study of Shah *et al.* (37), both studies indicated changes in zirconia properties caused by dipping.

Sedda *et al.* (33) evaluated the impact of coloring procedure on flexural resistance of zirconia blocks (industrially pre-colored blocks and green-stage infiltration-shaded blocks), and concluded that the tested coloring technique did not negatively influence the flexural strength of the tested zirconia. Though the present study assessed the μTBS rather than the flexural strength, a similar result was derived from the current research on coloring-induced changes in some tested groups. However, some studied groups showed dissimilarities compared to the study of Sedda *et al.* (33). This may be related to different coloring liquids and zirconia brands examined by the studies.

Hjerppe *et al.* (36) evaluated the effect of dipping in different coloring liquids on the biaxial flexural strength of zirconia and reported a negative effect of all tested coloring liquids except the coloring liquid D4. Though the present study assessed the bond of zirconia to cement, both studies revealed positive effects of coloring liquid D4 on zirconia properties.

The bond of zirconia to Panavia F2.0 cement is related to the bond between the metal oxides in zirconia ceramic and 10 hydroxydecyl dihydrogen phosphate (MDP) in Panavia F 2.0 (26, 29, 38, 39) .The phosphate group of MDP bonds to metal oxides of zirconia ceramic, while the vinyl group of MDP reacts with monomers in cement during polymerization (27). The metal oxides combination in the zirconia surface may impact the bond (27). This bond can be affected by some factors including surface treatment of zirconia ceramics (9, 12, 18), bonding technique (19, 21), aging (22-26), coloring (27), and Y-TZP particle deposition (28). In the dipping technique, coloring liquids with metal oxides may affect the bond by altering or replacing some metal oxides into zirconia surface microstructure. The current study results were varied among the tested coloring liquids (A3, B2, C1, D4) due to the coloring liquids specific compositions and their metal oxides.

Egilmez *et al.* (28) suggested a surface modification by Y-TZP particle deposition via dipping into the milling residue suspension before sintering in order to improve the bond between zirconia and resin cement. This surface modification was rather effective. Innovations in the bonding to zirconia-based materials have been developed by some researchers (15-21), but more investigations are needed to enhance the bond strength of zirconia to resin cement. As the coloring liquid D4 acted like an etching agent on zirconia and significantly increased the μTBS in this study, active ingredients of this liquid may be employed as a zirconia surface treatment. Among the examined color liquids in this study, only the coloring liquid B2 statistically compromised the bond of zirconia to resin cement. Therefore, improvement of this product is recommended to the manufacturer. Besides, dentists and dental technicians should apply this material under caution in cases with short crown height and low retention conditions.

These results are only limited to the materials and methods used in this study and therefore, they cannot be expanded to all types of zirconia, resin cements, and coloring liquids. Surface elemental analysis of examined zirconia and content analysis of coloring liquids are suggested for future studies.

Conclusion

Within the limitations of this study, it was concluded that depending on the shade of coloring liquids, dipping had positive, negative, or no effects on the μTBS of zirconia ceramic to Panavia F2.0 resin cement.

Conflict of Interest: ‘None declared’.

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