

# Effects of coloring procedures on shear bond strength between resin cement and colored zirconia

## Purpose

Debonding is expected as a frequent failure type in zirconia restorations. Therefore the aim of the current study is to evaluate the shear bond strength between colored zirconia and resin cement.

## Materials and methods

There were 11 groups evaluated each containing 12 zirconia discs (15 mm x 12 mm x 1.6 mm). Groups were colored with the colors A3, B1, C4, D2, and D4 of the VITA classical shade scale. Coloring procedure was carried out for either 3 second or 60 seconds for the study groups and the control group was left untreated. Specimens were then bonded to translucent resin cement having a thickness of 3 mm and width of 3 mm. The shear bond strength of the samples was measured in a universal testing machine with a crosshead speed of 1 mm per minute. Two-way analysis of variance and Tukey's HSD test were used for pairwise comparisons. Also paired t-test was used for comparing groups with the same color but having different shading times.

## Results

Any significant difference was not found between the shear bond strengths of samples depending on whether color or shading times. Among the groups, B1 (60 seconds of coloring) had the highest bond strength (10.05 MPa), while A3 (60 seconds of coloring) showed the lowest bond strength (6.72 MPa). However, these differences were not statistically significant.

## Conclusion

Coloring zirconia did not affect the shear bond strength between zirconia and resin cement.

**Keywords:** Shear bond strength; ceramic; coloring; zirconia; resin cement

## Introduction

Zirconia has a high fracture strength of more than 1000 MPa, fracture resistance of higher than 2000 N, and fracture toughness of 9–10 MN/m<sup>3/2</sup> (1). In addition to these favorable mechanical properties, it is chemically durable (2), biocompatible (3, 4), and displays esthetic advantages (5) over metal-ceramic restorations. However, recent clinical trials have shown that zirconia-based ceramic restorations' decementation is a common failure. Restoration's cementation corresponds to restoration durability, and cementation is also an important factor for marginal fit and fracture strength of restorations (6, 7). There are various cementation options for zirconia framework restorations. Cementation of zirconia restorations with traditional luting cements (such as glass ionomer or zinc phosphate cements) provides adequate clinical fixation, but adhesive cementation may be preferred for better retention and marginal adaptation (7–10). In addition, resin cement shows higher compressive and tensile strength than other luting agents, and allows color selection for more esthetic restorations (11, 12).

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Silica-based ceramics and resin cement show high bond strength with the use of hydrofluoric acid and silanization (13-15). Zirconia ceramics, however, exhibit high corrosion resistance, which inhibits acid etching. This limits their potential use with resin cements (16) and resin cementation of zirconia remains as a challenging subject on which various studies have been carried out. Although zirconia frameworks are more esthetic compared to metallic frameworks, their initial opaque and whitish appearance is still a handicap. As a result of this, colored zirconia frameworks were introduced to the market to obtain a more natural color. The main advantage of colored-zirconia ceramics is that they enable the selected color to be reflected from the inner layers of the restoration as in the dentin reflecting from inside of enamel (17). Zirconia frameworks can be colored with several techniques. The addition of metallic pigments to the initial zirconia powder (before or after milling blocks are pressed), the dipping of milled frameworks into the dissolved coloring agents, or the application of liner material to sintered frameworks are some of them (18).

There are few studies evaluating the effects of these color-shading procedures on the structure of zirconia-based restorations and shear bond strengths between zirconia and veneer ceramics. These studies has shown that the shear bond strength between zirconia and veneer ceramics is affected because coloring affects the structure of the zirconia framework (19-21). The aim of the current study was to evaluate the effects of different coloring liquids and different lengths of dipping times on the shear bond strength (SBS) between the zirconia framework and the resin cement. The null hypothesis is that dipping zirconia in different coloring liquids and for various dipping times does not affect the shear bond strength between zirconia and resin cement and the failure type is not affected from coloring.

## Materials and methods

### *Specimen preparation*

Zirconia blocks were partially stabilized with yttrium (ICE Zirkon, Zirkonzahn, South Tyrol, Italy) were cut into discs by means of a low-speed diamond saw (Struers Ltd., Lanarkshire, United Kingdom). The sizes of 132 discs were 15 mm x 12 mm x 1.6 mm. The samples were divided into eleven groups (n=12). Ten groups were colored with coloring liquids (Colour liquid, Zirkonzahn Inc., Norcross, GA, USA), and one control group was not colored. Five groups were colored with coloring liquids based on VITA shading (A3, B1, C4, D2, D4) for 3 seconds and five groups were colored with the same coloring liquids for 60 seconds by the aid of plastic holders. After the coloring procedure of the samples, except for the control group, they were dried under a warming lamp (Zirkonlampe 250, Zirkonzahn Inc., Norcross, GA, USA) for 45 minutes. Then the samples were sintered in a sintering oven (Zirkonofen 600, Zirkonzahn Inc., Norcross, GA, USA) according to the manufacturer's instructions.

After sintering, all samples were sandblasted with 50  $\mu\text{m}$  aluminum oxide ( $\text{Al}_2\text{O}_3$ ) particles (10 mm distance, 20 seconds duration, 3.5 atm pressure), in order to increase surface

roughness and enhance bond strength. Afterwards samples were cleaned in an ultrasonic cleaner (Quantrex 90, L&R Ultrasonics, Kearny, NJ, USA) for 10 minutes, rinsed and dried with air.

All samples were then treated with metal/zirconia primer (Ivoclar-Vivadent, Schaan, Liechtenstein) for 180 seconds prior to cement application. A PVC (polyvinyl chloride) ring (R-3603, Saint-Gobain S.A., Courbevoie, France) with an inner diameter of 3 mm and a height of 3 mm was positioned on the zirconia surface. Resin cement (Multilink Automix, Ivoclar Vivadent AG, Schaan, Liechtenstein) was mixed automatically in the syringe according to the instructions of the manufacturer and then applied to each surface of the zirconia samples by packing the inner cavity of the PVC ring (Figure 1).

Thirty minutes after irradiation, the rings around the cement cylinders were removed using a surgical blade by vertically cutting the ring into two or more fragments. Bonded samples were then stored in distilled water at 37°C for 24 hours.

### *Shear bond strength test*

The shear bond strengths (SBS) of the samples were measured at a speed of 1 mm per minute with a universal testing machine (TSTM 02500, Elista Ltd., İstanbul, Turkey) by an experienced observer (Figure 2). The accuracy of the load cell used was  $\pm 0.5\%$ . Following the SBS test, fracture modes were examined with an optical microscope (Olympus SZ4045 TRPT, Olympus Life Sciences, Tokyo, Japan) at magnifications of 10x and 20x, to determine types of failure. Potential fracture types were classified as cohesive, adhesive, or combined (a category which includes both cohesive and adhesive fractures).

### *Statistical analysis*

Statistical analysis of the data was performed with Statistical Package for Social Sciences (SPSS) statistical software (SPSS PC, Vers.15.0; SPSS Inc.; Chicago, IL, USA). As all the variables were numerical and the distribution of the data met the assumptions for normality, two-way analysis of variance (ANOVA) was used to compare multiple groups having the same shading times, followed by Tukey's Honestly Significant Difference (HSD) post-hoc test for pairwise comparisons. A paired t-test was used for groups of the same color but different shading times. Confidence interval was set to 95% and p values less than 0.05 were considered significant.

## Results

The SBS results of the samples, according to different color shades and coloring times, are given in Table 1. The B1 group, which was colored for 60 seconds, showed the highest SBS value of 10.05 MPa. The A3 group, colored for 60 seconds, showed the lowest SBS value of 6.72 MPa. The non-colored control group showed the second-highest SBS value, at 9.35 MPa. However there was no significant difference between groups. All fractures between the zirconia framework and the resin cement were adhesive. Cohesive or combined fractures were not observed.

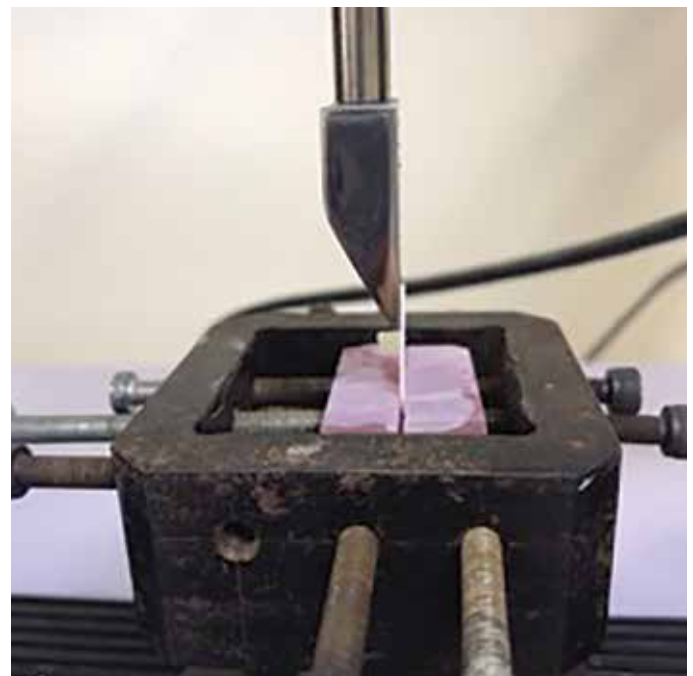
**Table 1.** Mean shear bond strengths of the groups with standard deviations

Groups	Mean (MPa)	SD ( $\pm$ )	Min (MPa)	Max (MPa)
A3 (3 s)	7.15	3.07	4.33	15.89
A3 (60 s)	6.72	1.67	5.67	10.48
B1 (3 s)	7.68	1.38	5.09	10.45
B1 (60 s)	10.05	4.26	5.87	21.14
C4(3 s)	8.68	1.87	6.70	12.55
C4 (60 s)	8.68	2.80	6.42	14.79
D2(3 s)	8.51	3.42	5.49	16.71
D2 (60 s)	8.14	1.64	6.68	12.55
D4(3 s)	8.05	2.40	5.45	12.54
D4 (60 s)	8.49	2.73	6.42	15.21
Control	9.34	2.61	6.51	15.92

MPa: megapascal; SD: standard deviations



**Figure 1.** A test sample with 3 mm thick resin layer before being subjected to shear bond strength testing.



**Figure 2.** The test set-up for shear bond strength testing.

## Discussion

The coloring procedures have been used for several years, however, the effect of coloring procedures on the bond strength between zirconia frameworks and resin cement has not been investigated. The results of the current study have shown that the SBS between the zirconia framework and the resin cement is not affected by either the coloring shade or the dipping time; so the null hypothesis is accepted for both SBS and fracture type.

Previous studies investigating the SBS of veneer ceramic with colored zirconia have reported that coloring procedures can affect the results (20, 22, 23). Chevalier *et al.* (24) reported that concentration of coloring pigments at grain boundaries reduces the percentage of the stabilizing element (yttrium). Reduction in the percentage of the stabilizing element would likely result in a higher frequency of tetragonal-monoclinic phase transformation, which would affect the mechanical

properties of the zirconia. As a further explanation of the mechanical effects of coloring, Chen and Chen (25) have explained that the melting point of the coloring pigment (2410°C) is much lower than the melting point of yttrium and hafnium oxides (2751°C). Therefore, displacement of the stabilizing elements by the metallic pigments can occur during the sinterization of zirconia frameworks (2).

A previous study reported that light-cure resin cements and dual-cure resin cements, which are activated by a light source, cannot be sufficiently polymerized if light cannot penetrate through the material (26-28). In a recent study, Heffernan *et al.* (29) compared the translucency of different all-ceramic core materials, finding that In-ceram zirconia has the highest opacity (with a 1.00 contrast ratio) when compared to the same-value metal-ceramic specimens. Therefore, dual-cure resin cements are more reliable for low-translucency ceramics, and this study focused on a certain dual-cure resin cement only.

In the current study, the SBS values obtained were higher than those reported by Moon *et al.* (30) who had used the same resin cement and a metal/zirconia primer. However, the SBS results of the current study were lower than studies which have used resin cements based on MDP monomer (31-35). The absence of adhesive functional monomers in the resin cement may explain the lower SBS values and adhesive failure at the zirconia-resin cement interface when compared to resin cements based on MDP monomer (14, 36).

In this study, the SBS between zirconia and resin cement ranged from 6.72 MPa to 10.05 MPa. Luthy *et al.* (15) reported that a minimal bond strength of 10–13 MPa is required for an acceptable clinical bonding. The present study demonstrated a SBS lower than this clinically acceptable threshold for all groups. It should also be noted that, although coloring procedures do not affect the shear bond strength between zirconia and the resin cements used in the current study remains clinically unacceptable according to Luthy *et al.* (15).

The present study demonstrates that the coloring procedure and duration applied to the zirconia framework has no effect on the SBS of the resin cement. However, the study is limited to a single resin cement (Multilink) and a single zirconia framework system (Zirkonzahn). A future objective is to measure the effects of different types of resin cements on the SBS of other zirconia systems.

## Conclusion

Different coloring liquids and dipping times do not affect the shear bond strength between the zirconia framework and resin cement. Adhesive type of fractures were observed for all samples. Still, the shear bond strength values of non-MDP containing resin cement used in this study are not enough for clinical use for both colored and non-colored zirconia frameworks.

**Ethics Committee Approval:** Ethics committee approval was not needed for the current *in vitro* study.

**Informed Consent:** Not required.

**Peer-review:** Externally peer-reviewed.

**Author Contributions:** İT designed the study. İT and İT generated, gathered and analyzed the data. İT wrote the majority of the original draft. İT participated in writing the paper. All authors approved the final version of the paper.

**Conflict of Interest:** The authors have no conflicts of interest to declare.

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**Türkçe Öz:** Renklendirme süreçlerinin zirkonya ile rezin siman arasındaki bağlanma dayanımı üzerindeki etkileri. Amaç: Desimantasyon zirkonya destekli restorasyonlar için sık görülen sorunlarından biridir. Bu çalışmanın amacı, renklendirilmiş zirkonyum oksit alt yapı ile rezin siman ile arasındaki makaslama bağlanma dayanımını incelemektir. Gereç ve Yöntem: Her birinde 12 zirkonya disk (15 mm x 12 mm x 1.6 mm) olacak şekilde 11 grup oluşturulmuştur. Gruplar VITA klasik

renk skalasına göre: A3, B1, C4, D2 ve D4 renklerine uyumlu olarak renklendirilmiştir. Tüm gruplara önerilen 3 saniyelik renklendirme ve 60 saniyelik uzatılmış renklendirme işlemi uygulanmıştır. Kontrol grubunda renklendirme yapılmamıştır. Örneklere 3 mm kalınlığında ve 3 mm yüksekliğinde translusent rezin siman uygulanmış ve üniversal test cihazında 1 mm/dakika hızında bağlanma dayanımı testi yapılmıştır. İki yönlü varyans analizi (ANOVA) ve ikili karşılaştırmalarda Tukey HSD testi kullanılmıştır. Aynı renkte olan ancak farklı tonlama süresine maruz kalan örnekler ise t-testi ile değerlendirilmiştir. Bulgular: Makaslama bağlantı dayanımının renk ya da renklendirme sürelerine bağlı olarak anlamlı bir fark göstermediği belirlenmiştir. 11 araştırma grubunun içinde en yüksek bağlantı değeri (10,05 MPa) 60 saniye renklendirme uygulanan B1 renk grubunda; en düşük bağlantı değeri (6,72 Mpa), 60 saniye renklendirme uygulanan A3 renk grubunda elde edilmiştir. Ancak bu farklar istatistiksel olarak anlamlı değildir. Sonuç: Zirkonya alt yapı ve rezin siman arası bağlantı dayanımı zirkonyayı renklendirme işlemlerinden etkilenmemektedir. Anahtar kelimeler: Makaslama bağlanma dayanımı; seramik; renklendirme; zirkonya; rezin siman

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