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RESEARCH ARTICLE

EFFECT OF LASER AND SURFACE TREATMENTS ON BOND STRENGTH OF LUTING AGENTS TO COLORED AND NON-COLORED ZIRCONIA

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ABSTRACT

The purpose of this study was to evaluate the effects of different surface treatments and zirconia type (white or colored) on shear bond strength (SBS) between zirconia and resin cements. Two main groups were created according to zirconia color. After coloring, white and colored sintered zirconia was divided into four surface treatment groups: control, air abrasion, silica coating, and air abrasion + erbium-doped yttrium-aluminum-garnet (Er:YAG) laser (n=20). After surface treatment, surface roughness and scanning electron microscopic analyses were conducted. Then, Panavia F 2.0 and RelyX Ultimate Clicker cements were each applied to 10 specimens from each subgroup. The specimens were stored in distilled water at 37°C for 24 h, and then subjected to 5,000 thermal cycles between 5°C and 55°C. SBS was measured using a universal testing machine at a crosshead speed of 0.5 mm/min. SBS values were compared using analysis of variance and Turkey's honestly significant difference test ($p < 0.05$). Roughness values were analyzed using ordinary linear regression. SBS was significantly greater in the air abrasion + Er:YAG laser group (14,055 MPa), and the control group (5,678 MPa) had the lowest value; it did not differ between the air abrasion and silica coating groups. SBS of colored specimens (9, 33 MPa) was significantly lower than that of white specimens (10,45 MPa). Roughness was related significantly to bond strength. These results show that all tested surface treatments were suitable, but that air abrasion + Er:YAG laser treatment was most effective.

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INTRODUCTION

The demand for high-strength esthetic dental materials has prompted the development of all-ceramic systems (Cavalcanti *et al.,* 2009). Due to its good mechanical properties and with advances in computer-aided design/computer-aided manufacture (CAD/CAM) technology, zirconia is used for the production of fixed dental prostheses, posts-cores, and implant abutments (Kwon *et al.,* 2013). Zirconia restorations are more esthetic than metal-ceramic restorations; although zirconia ceramic is overly white, colored zirconia ceramics have been introduced to improve overall color matching results (Ardlin, 2002). Zirconia ceramics can be shaded using various techniques, such as the addition of metallic pigments to the milling blocks, the dipping of milled frameworks into dissolved coloring agents, and the application of liner material to the sintered white frameworks (Aboushelib *et al.,* 2005). The clinical success of ceramic restorations depends on not only their mechanical properties. Resistant and durable bonds between ceramics and resin cements are fundamental for the long-term success (Burke *et al.,* 2002). Zirconia ceramics can be cemented using conventional and adhesive techniques (Palacios *et al*., 2006). However, the use of a resin cement improves retention, fracture resistance, and marginal adaptation of the restoration (Burke *et al.,* 2002). The use of adhesive cements containing phosphate monomer (MDP) may contribute to the bonding of zirconia restorations. Because of the lack of silica and a glass phase, conventional adhesive conditioning methods, such as etching with hydrofluoric acid and silanization, are not efficient for zirconia ceramics (Luthy *et al.,* 2006). For this reason, alternative surface treatment methods are necessary. Roughened ceramic surfaces may allow resin cements to flow into microretention spaces, creating resistant micromechanical interlocking (Kern *et al.,* 1998). Surface treatment methods, such as air abrasion with aluminum oxide (Al_2O_3) and coating with silica-modified Al_2O_3 particles, have been introduced for zirconia ceramics (Kern *et al.,* 1998; Piwowarczyk *et al.,* 2005). In addition, laser-induced modification of zirconia has also been investigated (Shiu *et al.,* 2007). However, the most appropriate surface pretreatment technique for zirconia remains unclear.

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This study aimed to evaluate the effects of surface treatment on the surface morphology of zirconia and to investigate the bond strength of MDP-containing resin cements to zirconia after different surface and coloring treatments. The following null hypotheses were tested: (1) the type of zirconia (white or colored) and surface treatment will not affect shear bond strength at the cement–zirconia interface, (2) surface treatment will not affect the surface roughness of zirconia, and (3) air abrasion+erbium-doped yttrium-aluminum-garnet (Er:YAG) laser treatment or silica coating will not affect the bond strength of zirconia.

MATERIALS AND METHODS

Specimen preparation

A yttria-stabilized tetragonal zirconia (Y-TZP) ceramic (In-Ceram YZ for inLab®, VITA Zahnfabrik, Bad Säckingen, Germany) was used. Specimens (n=160; 8.75×8.75 mm) were produced from presintered blocks using a CAD/CAM system (Yena D40, Yenadent, Istanbul, Turkey) and were divided into two main groups (n=80 each) according to type of zirconia: white (no coloring) and colored (In-Ceram YZ Coloring Liquid, VITA Zahnfabrik, Bad Säckingen, Germany). They were sintered to a final dimension of 7×7 mm in a furnace (Zyrcomat, VITA Zahnfabrik, Bad Säckingen, Germany) at 1530°C for 7.5 h, in accordance with the manufacturer's instructions. All specimens were smoothed with 600-, 800-, and 1200-grit silicon carbide papers (English Abrasives, England) for 15 s using a 300-r/min polisher under water irrigation (LaboPol-5, Struers, Denmark) to obtain a standardized surface polish. After polishing, all specimens were cleaned ultrasonically (Sonorex RK102, Bandelin, Walldorf, Germany) in distilled water. Each specimen was embedded in an autopolymerizing acrylic resin block with one ceramic surface exposed. The specimens were divided into four groups (n=40) according to surface treatment (Table 1). After surface treatment, the samples were cleaned ultrasonically in 99.6% acetone (ZAG Kimya, Istanbul, Turkey) for 5 min, and then in distilled water for another 5 min (Yang B *et al.,* 2007).

Surface roughness evaluation

After the surface treatments, the average roughness (Ra) of each specimen was measured using a surface profilometer (Surtronic 25, Taylor Hobson, Leicester, England), with a cutoff value of 2.40 mm and measurement length of 0.8 mm. Four measurements taken at different locations were recorded for each specimen. The average was calculated to obtain the Ra value.

Scanning electron microscopic examination

One specimen selected randomly from each group was analyzed using a scanning electron microscope (SEM, JSM-5600, JEOL Ltd., Tokyo, Japan) at 20 kV. An image from each surface was taken at ×1,000 magnification.

Bonding

Each group was divided into two subgroups (n=10) according to resin cement: group P (Panavia F2.0, Kuraray, Osaka, Japan) or group Rx (RelyX Ultimate Clicker, 3M ESPE, Seefeld, Germany). Teflon tubes with internal diameters of 4 mm and heights of 2 mm were placed on the specimens and filled with resin cement.

In group P, an MDP-containing primer (Clearfil, Kuraray, Osaka, Japan) was applied to the specimen surfaces and left to dry for 5 min. Equal amounts of Panavia F 2.0 pastes A and B were mixed for 20 s and inserted into the plastic mold. The resin cement was light polymerized for 40 s (Elipar S10 LED, 3M ESPE, Seefeld, Germany) from the top and each side, for a total of 200 s. The cement surface was protected with an oxygen barrier (Oxyguard II, Kuraray, Osaka, Japan) for 3 min. In the Rx group, Single Bond Universal Adhesive (3M ESPE, Seefeld, Germany) was applied to the surfaces and left to dry for 20 s. Equal amounts of RelyX Ultimate Clicker pastes A and B were mixed for 20 s. The resin cement was light polymerized as described above. All specimens were stored in distilled water at 37°C for 24 h. They were subjected to 5,000 thermal cycles between 5°C and 55°C (DTS B1 Dentester, Salubris Technica, Istanbul, Turkey), with a transfer time of 2 s and dwell time of 30 s.

Shear bond strength test

Shear bond strength was tested using a universal testing machine (Instron 3345, Norwodd, USA) at a crosshead speed of 0.5 mm/min until failure occurred. The fractured surfaces of all specimens were assessed under a stereomicroscope at ×32 magnification. Fracture patterns were classified as reflecting adhesive failure (resin cement on <20% zirconia ceramic), cohesive failure (resin cement on >80% zirconia ceramic), and mixed failure (resin cement on 20–80% zirconia ceramic) (Usumez A *et al.,* 2013).

Statistical analysis

Statistical analyses were performed using SPSS software (ver. 15.0 for Windows, SPSS Inc., Chicago, IL, USA). Data from the different groups were analyzed using three-way analysis of variance (ANOVA). Multiple comparisons were performed using Tukey's honestly significant difference test. Ra values were analyzed using ordinary linear regression. All statistical analyses were performed with a significance level of p=0.05.

RESULTS

Surface roughness

Linear regression analysis showed that mean Ra values were significantly higher in the air abrasion+Er:YAG group (1.49 µm) than in the other surface treatment groups (p<0.05; Table 2). The control group had the lowest Ra value (0.28 µm; p<0.05). Ra values did not differ significantly between the air abrasion group (0.95 µm) and the silica coating group (0.65 µm). One-way ANOVA revealed no significant difference in Ra values according to zirconia color (Table 3, Fig. 1).

Surface morphology

SEM examination showed smoother surface profiles in the control group (Fig. 2). Sandblasting with Al_2O_3 particles created clearly rougher surfaces, with the formation of microretentive grooves and pits (Fig. 2b). Silica-coated specimens showed micromechanical surface irregularities and a thin, microretentive layer (Fig. 2c). Air abrasion+Er:YAG laser treatment produced deep pits and irregular microcracks (Fig. 2d).

Table 1. Surface treatments

Group	Surface treatment
Control	None
Air abrasion	Air abrasion with 110-µm Al_2O_3 particles (Korox 110; Bego, Bremen, Germany) for 15 s under 2.8 bar pressure at a distance of 10 mm
Silica coating	Air abrasion with 30-µm silica-coated Al_2O_3 particles (Cojet Sand; 3M ESPE, Seefeld, Germany) for 15 s under 2.8 bar pressure at a distance of 10 mm
Air abrasion + Er:YAG laser	Air abrasion with 110-µm Al_2O_3 particles for 15 s under 2.8 bar pressure at a distance of 10 mm. Er:YAG laser (AT Fidelis Er:YAG; Fotona, Ljubljana, Slovenia) application at a wavelength of 2.940 nm using an H14 handpiece with an optical fiber diameter of 1.2 mm. Er:YAG laser parameters: energy, 400 mJ; pulse rate, 10 Hz; power, 4 W; MSP mode pulse width, 100 µs for 15 s at a distance of 1 mm

Table 2. Surface roughness of zirconia ceramics according to surface treatment

Group	Surface roughness (Ra, µm) p<0.05
Control (Co)	0.28 (0.13)
Air abrasion (A)	0.95 (0.22)
Silica coating (S)	0.65 (0.25)
Air abrasion + Er:YAG laser (AL)	1.49 (0.39)

Data are presented as mean (standard deviation).

Table 3. Results of one-way ANOVA of surface roughness according to zirconia color

	Df	Sum Sq.	Mean Sq.	F	p
Groups (W-C)	1	0.058	0.0583	0.276	>0.05
Residuals	118	24.919	0.2112		

Table 4. Shear bond strength (MPa) of zirconia ceramic to resin cement according to surface treatment

Zirconia color	Surface treatment	Panavia F 2.0	RelyX Ultimate Clicker	p
White	Air abrasion	9.66 (1.22)	11.63 (1.43)	<0.001
	Silica coating	7.37 (2.49)	13.25 (1.58)	<0.001
	Air abrasion + Er:YAG laser	16.31 (2.07)	14.71 (1.1)	<0.001
	Control	5.34 (1.34)	5.35 (1.09)	>0.05
Colored	Air abrasion	10.73 (2.18)	9.94 (1.96)	>0.05
	Silica coating	6.12 (0.52)	10.67 (1.54)	<0.001
	Air abrasion + Er:YAG laser	13.78 (2.47)	11.40 (1.50)	<0.001
	Control	5.58 (1.11)	6.43 (1.32)	<0.001

Data are presented as mean (standard deviation)

Table 5. Distribution of failure modes

Group	Panavia F 2.0			RelyX Ultimate Clicker		
	A	C	M	A	C	M
White, air abrasion	30%	-	70%	20%	-	80%
White, silica coating	10%	-	90%	40%	-	60%
White, air abrasion + Er:YAG laser	20%	20%	60%	40%	-	60%
White, control	100%	-	-	90%	-	10%
Colored, air abrasion	30%	-	70%	20%	10%	70%
Colored, silica coating	40%	-	60%	20%	-	80%
Colored, air abrasion + Er:YAG laser	30%	20%	50%	20%	-	80%
Colored, control	90%	-	10%	100%	-	-

A, adhesive; C, cohesive; M, mixed.

Bond strength and failure

Three-way ANOVA showed that shear bond strength was affected by surface treatment (F=95.69), zirconia type (*white or colored) (F=10.124), and cement type (F=9.048; all p<0.001). Shear bond strength was significantly greater in the air abrasion+Er:YAG laser group than in the other surface treatment groups (p<0.05; Table 4). The shear bond strength of the air abrasion group (9.357±3.26 MPa) was significantly greater than that of the control group (5.678±1.25 MPa), but did not differ significantly from that of the silica coating group (10.494±1.84 MPa) and groups did not differ. Regardless of surface treatment and zirconia type, bond strength was significantly greater in Rx than in P specimens (p<0.05). However, white zirconia treated with air abrasion+Er:YAG laser in group P had the greatest shear bond strength. According to Student's t test, shear bond strength was greater in white zirconia specimens than in colored specimens (p<0.05). The modes of failure are summarized in Table 5. The predominant failure type was mixed (53%), followed by adhesive failure (44%) and cohesive failure (3%; Fig. 3) (Usumez A *et al.,* 2013).

DISCUSSION

This in vitro study evaluated surface changes in zirconia after different surface treatments and shear bond strength of zirconia to MDP-containing resin cements after coloring and aging.

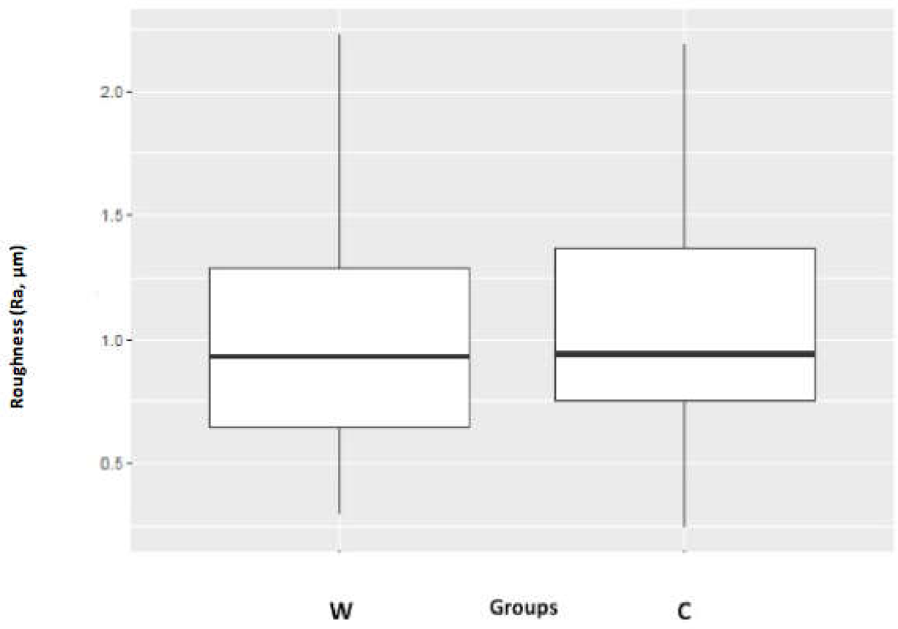

Fig. 1. Relation between coloring and surface roughness

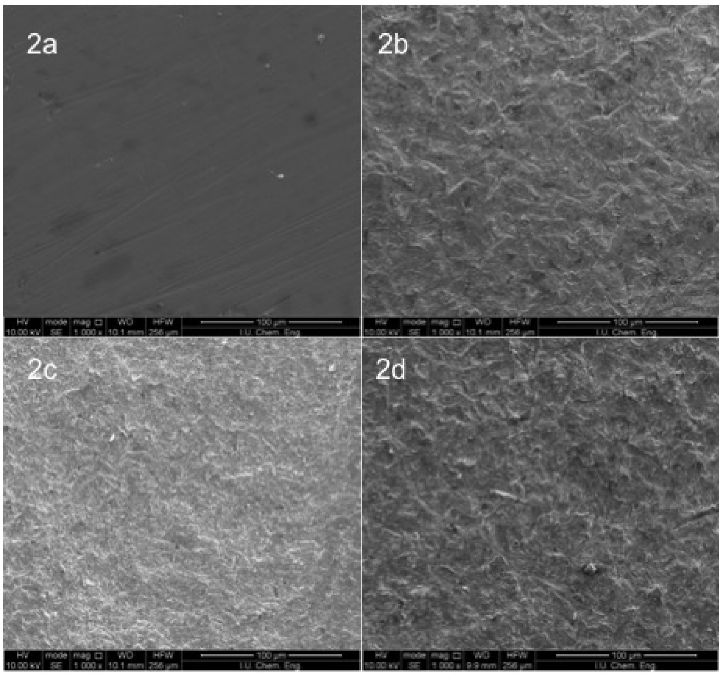

Fig. 2. SEM images of zirconia surfaces. (a) Control, (b) air abrasion, (c) silica coating, (d) air abrasion + Er:YAG laser (×1000 magnification)

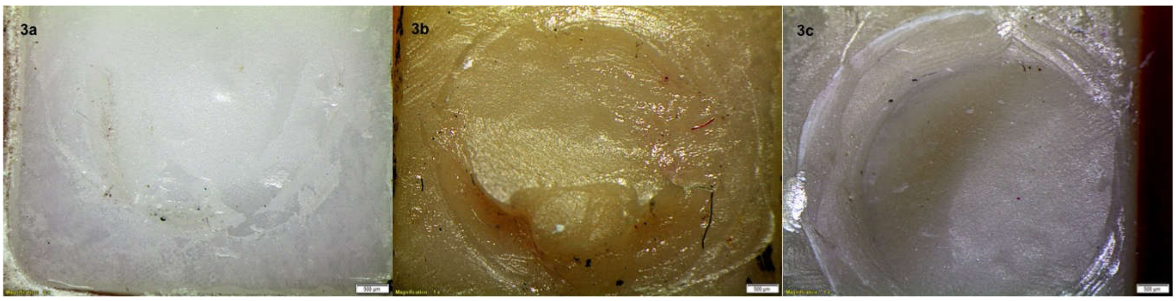

Fig. 3. Stereomicroscope images of different failure modes. (a) Adhesive, (b) cohesive, (c) mixed (×32 magnification)

From the results of this in vitro study, the all three null hypotheses were rejected, as bonding effectiveness was greater for white than for colored zirconia, mechanical and chemical surface treatments increased surface roughness, and air abrasion+Er:YAG laser treatment increased the shear bond strength of resin cement to the zirconia surface. Surface roughness is an important factor for adhesion. A rough surface increases the surface area, facilitates wettability by reducing surface tension, and creates micromechanical retention. Successful bonding between ceramic restorations and resin cement requires chemical and micromechanical retention, and the results of this study indicate that the surface treatment method is the most important factor affecting bonding (Ozcan M *et al.,* 1998). Air abrasion and silica coating were applied based on previous studies, and the laser parameters were selected from a previous pilot study (Nothdurft FP *et al.,* 2008; Subasi MG *et al.,* 2014). Sandblasting is the preferred method of modifying zirconia surfaces, and the use of Al_2O_3 particles produces abundant hydroxyl groups on the surface, providing micromechanical retention (Amaral R *et al.,* 2006; Ozcan M *et al.,* 2003). According to the results of this study it can be said that surface treatment methods are the most effective factor on resin cementation of zirconia. All treatments were used to provide chemical and micromechanical retention on the zirconia surface. Air abrasion and silica coating were applied base on the literatures, and the laser parameters were selected from previous pilot study (Nothdurft *et al.,* 2008; Subasi *et al.,* 2014).

Sandblasting is the most preferred surface treatment method to modify surface of zirconia (Amaral R *et al.,* 2006; Ozcan M *et al.,* 2003). When zirconia surface exposed the sandblasting with Al_2O_3, this process results more hydroxyl groups formed at the surface and so micromechanical retention is provided (Ozcan M *et al.,* 2003). In the present study, Al_2O_3 air abrasion increased the surface roughness and shear bond strength of resin cement to zirconia compared with the control group in this study, in agreement with the results obtained by Kern et al (Kern M *et al.,* 2009). SEM images of the air abraded group were apparent retentive pits and scratch-like lines. The tribochemical silica coating of zirconia has been found to effectively increase bond strength to adhesive cements. In silica coating treatments, air abrasion induces micromechanical bonding, and chemical adhesion is achieved by a silica layer consisting of silane (Ozcan M *et al.,* 2003). The microretentive layer observed on silica-coated surfaces in this study may have increased bond strength to the resin cement, but shear bond strength did not differ between the air abrasion and silica coating groups. The Cojet system (9.357±3.26 MPa) resulted in higher SBS values than control (5.678±1.25 MPa) in this study, but sandblasting with 30 μm silica-coated alumina showed similar results as Al_2O_3 (10.494±1.84 MPa). The use of laser etching for surface roughening is an alternative and innovative method. Several studies have shown that Er:YAG laser treatment of zirconia creates rough surfaces (Kasraei S *et al.,* 2014; Lin Y *et al.,* 2013).

They reported that Er:YAG laser treatment showed a rough surface pattern on zirconia. In the present study, air abrasion+Er:YAG laser treatment produced the greatest shear bond strength and roughness. All groups except the control group showed effective bonding, according to Behr et al.'s definition of clinically acceptable bond strength as >10 MPa, confirming the importance of surface treatment of zirconia ceramics for bonding to resin cement (Behr M *et al.,* 2011).

To obtain adequate bond strength between zirconia and adhesive resin, creation of mechanical bond through surface roughening and chemical bond by use of functional monomers is essential (Magne P *et al.,* 2010). There is a reaction between Phosphate ester monomers, such as MDP, react chemically with zirconia, ensuring durable and water-resistant chemical bonding and increasing bond strength between treated zirconia and adhesive resin (Magne P *et al.,* 2010; Tzanakakis EGC *et al.,* 2016). Turp et al. reported that the presence of MDP in any component of the bonding/silane/resin cement complex significantly increased bond strength between zirconia and resin cement (Turp V *et al.,* 2016). In this study, two MDP-containing resin cements were used to evaluate the efficiency of MDP monomer after aging. Compared with RelyX Ultimate Clicker, Panavia F 2.0 showed increased bond strength in the air abrasion+Er:YAG laser group, but not in the silica-coated group. Thus, the use of RelyX Ultimate Clicker on silica-coated zirconia is recommended to improve long-term bonding. As a result of the present study, zirconia coloring process significantly decreased shear bond strength, as the composition of the liquid shade led to increased porosity (compromising mechanical properties) and the addition of pigment changed the chemical structure of zirconia. Mosharraf et al. evaluated the effects of different surface treatments and colored zirconia frameworks on bond strength to zirconia and surface treatment had a significant effect on the bond strength between zirconia and ceramic, while using colored frameworks had no effect (Mosharraf R *et al.,* 2011). Hjerppe et al examined the effects of different coloring solutions and application times on the fracture resistance (biaxial flexural strength) of zirconia, and found that the coloring procedure had a negative effect, whereas the D4 shade had a positive effect (Hjerppe J *et al.,* 2008). Similarly, Mashid et al. showed that the D4 liquid shade positively affected bond strength, whereas the B2 and C1 shades decreased bond strength in comparison with the control group and the A3 shade had no effect on the bond strength of zirconia to adhesive resin cement (Mahshid M *et al.,* 2015). Some study findings suggest that the bonding mechanism of zirconia to resin cement is related mainly to the bond between the metal oxides in Y-TZP ceramic and MDP in MDP-containing resin cement (Luthy H *et al.,* 2006). The phosphate group of MDP bonds strongly to these metal oxides, and the vinyl group of MDP reacts with monomer with resin cements when the resin is polymerized. These oxides are added to zirconia during manufacturing and coloring (Ardlin BI, 2002). When zirconia is dipped in liquid shades containing metal oxides before sintering, some of these oxides may infiltrate the zirconia surface microstructure. The composition of liquid color shades may thus be responsible for positive, negative, and neutral effects.

Chemical, mechanical, and thermal factors in the mouth may affect the adhesion of ceramic to cement. In this study, a standardized electronic thermal cycling device was used according to the ISO/TS 11405 recommendations (ISO TS 11405). Other studies reported that the bond strength values after aging seen a significant reduction (Kern M *et al.,* 1998; Ozcan M *et al.,* 2015). Common consensus on aging procedures and parameters used in studies of effects on bonding is needed. Until such consensus is achieved, the use of at least 5,000 thermal cycles, as in the present study, has been recommended (Ozcan M *et al.,* 2015). This study had several limitations. Thermal aging procedures were applied to all samples, with no testing of its effects. We could not determine the difference of thermal cycling was applied and not applied.

Therefore, to determine the effects of thermal aging on bond strength, samples not subjected to thermal cycling should be added in future studies.

Conclusion

Within the limitations of this study, the following conclusions were drawn:

- Surface treatment, zirconia type, and cement type affected the bond strength of resin cement to zirconia, with surface treatment showing the strongest effect.
- All surface treatments increased surface roughness, with the highest Ra values (and greatest shear bond strength) observed in the air abrasion + Er:YAG laser treatment group. No difference in Ra values was observed between the air abrasion and silica coating groups.
- The coloring process changed the chemical structure of zirconia and decreased shear bond strength.
- MDP monomer–containing cements were suitable for use with zirconia ceramics, but RelyX Ultimate Clicker yielded significantly better results than did Panavia F 2.0.
- Panavia F 2.0 was less effective than RelyX Ultimate Clicker for silica-coated zirconia surfaces, but it improved shear bond strength compared with the control group.

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