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Fracture Resistance, Surface Roughness, and Microtensile Bond Strength of Monolithic Zirconia to Resin Cements after Plasma Treatment

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A B S T R A C T

Objectives: This study aimed to assess the effect of surface treatment with plasma on surface roughness (SR) and fracture resistance (FR) of monolithic zirconia, and its microtensile bond strength (MTBS) to resin cements.

Materials and Methods: This in vitro, experimental study was conducted on 40 monolithic zirconia crowns for FR test, 100 zirconia rods for MTBS test, and 40 zirconia blocks for profilometry. According to the surface treatment type, the samples were randomly assigned to 4 groups of (I) control (no surface treatment), (II) argon-oxygen plasma (AOP), (III) argon plasma (AP), and (IV) sandblasting (SB). FR of crowns and MTBS of zirconia rods to Allcem Dual and Panavia SA resin cements were measured by a universal testing machine, surface texture was evaluated by atomic force microscopy (AFM), and SR was measured by a profilometer. Data were analyzed by one-way and two-way ANOVA, Tukey's test, and independent t-test (alpha=0.05).

Results: There was a significant difference in SR among the groups (P=0.003). The AP group had significantly lower SR than other groups (P=0.01). FR was not significantly different among the four groups. The MTBS in the SB and AOP groups was significantly higher than that in the control and AP groups for both resin cements. MTBS was not significantly different between the two resin cements within each group.

Conclusion: None of the surface treatments affected the FR of zirconia crowns. AOP and sandblasting techniques increased the MTBS of zirconia to resin cements with unnoticeable change in SR.

Keywords: Plasma Gases; Resin Cements; Zirconium

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INTRODUCTION

Advances in the computer-aided design/computer-aided manufacturing (CAD/CAM) technology in the recent years have led to improvements in accuracy and marginal adaptation of indirect restorations and their faster fabrication. Also, different materials such as glass ceramics, zirconia ceramics, and hybrid ceramics have been introduced for the fabrication of indirect restorations [1].

The conventional 3 mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) is infrequently used in anterior teeth due to its opaque appearance and the possibility of ceramic veneer chipping [2]. Monolithic full-contour zirconia containing 4 or 5 mol% yttria was introduced to the market for this purpose. Increasing the concentration of yttria is the best method to increase the translucency of zirconia. It increases the cubic phase, which is optically

isotropic, and decreases the tetragonal phase, and subsequently reduces light refraction [3]. However, it has different mechanical properties compared with 3Y-TZP zirconia [4].

To date, several methods have been proposed to enhance the bond strength of zirconia substrate to resin materials, such as air abrasion with aluminum oxide particles (Al_2O_3), chemical methods such as the application of materials containing functional monomers like 10-MDP, laser surface treatment, and tribochemical coating [5].

Sandblasting of 3Y-TZP zirconia surface with Al_2O_3 is a commonly practiced method to achieve a durable bond between the ceramic and resin. Sandblasting increases the surface roughness (SR) and contact area, and enhances the mechanical interlocking of resin in the ceramic surface. It can also increase the strength of conventional zirconia by phase transformation toughening. Nonetheless, highly translucent zirconia does not undergo phase transformation, and sandblasting can cause micro-cracks and damage the surface of reduced strength monolithic zirconia [6,7].

Dental applications of plasma as a novel surface treatment have increased in the recent years. Surface treatment with plasma is generally used for cleaning and activation of surface, and to increase surface wettability and surface energy and enhance the bond strength as such. The non-thermal cold plasma gas is ionized to the level that generates highly reactive particles such as O_3, OH, H_2O_2, NO, OH, and singlet oxygen in high amounts at low temperatures, which can change the non-reactive functional groups on the zirconia surface to reactive radicals that participate in bonds [8,9].

The efficacy of plasma for bond strength enhancement of 3Y-TZP dental zirconia to resin cements has been previously studied. Canullo et al, [10] and Ito et al. [11] demonstrated that argon plasma increased the bond strength of zirconia to resin cement. On the other hand, Balkenhol et al. [12] reported that surface treatment with atmospheric plasma had inconsistent effects on bond strength, and they did not recommend it for use in the clinical setting. Kim et al. [13] stated that the surface energy of Y-TZP increased when treated with plasma, but bond strength to resin was not significantly improved. Studies on the effects of plasma on bond strength and fracture resistance of monolithic translucent zirconia are limited. Thus, the aim of this study was to investigate the effect of plasma of argon, oxygen and a mixture of both on fracture resistance (FR) and microtensile bond strength (MTBS) of monolithic zirconia to resin cements. The null hypotheses of the study were: (I) plasma treatment would not affect the FR of monolithic zirconia, and (II) the MTBS of monolithic zirconia to resin cements would be independent of the type of surface treatment and type of cement.

MATERIALS AND METHODS

The protocol of this in vitro study was approved by the ethics committee of Babol University of Medical Sciences (IR.MUBABOL.HRI.REC.1400.222).

Fabrication of metal die:

The surface of an acrylic model of maxillary first premolar with jacket crown preparation (A25A UL41, Nissin Dental Products, Kyoto, Japan) was scanned by a 3D scanner (I3Dscan eco, imes-icore, Hessen, Germany), and the scan file was transferred to a CAD/CAM system software (CORiTECH 250i, imes-icore Hessen,Germany). Next, the design of a premolar tooth with a standard full-ceramic crown preparation was transferred to a CAD machine, and 40 acrylic cores were milled from polymethyl methacrylate discs (Yamahachi Dental MFH, Gamagori, Japan). Acrylic specimens were then invested, burnt-out, and cast with base-metal alloy (VeraBond, Aalba Dent, Fairfield,USA). Accordingly, 40 metal dies as master metal dies were obtained for the fabrication of zirconia crowns.

Fabrication of zirconia crowns:

A CAD/CAM machine (CORiTECH 250i, imes-icore, Hessen, Germany) was used for the fabrication of zirconia crowns. First, scan spray was sprayed on the surface of metal dies to create contrast, and then their surface was scanned by a 3D scanner. Using the device software, a full-contour crown was virtually designed such that the zirconia crown thickness was 1 mm at the margin, and 1.5 mm at the occlusal surface and axial walls. This design was transferred to a CAM machine, and 40 zirconia crowns were milled from 5Y-PSZ monolithic zirconia

material (Luxen Smile, SMHT; DENTALMAX, Seoul, Korea). The list of materials that were used in this study is provided in Table 1. Zirconia crowns were sintered in a furnace (Auto sinter 1650; KFP Dental, Tehran, Iran) at 1500°C according to the sintering schedule recommended by the manufacturer for 10 hours. The coefficient of contraction of zirconia was considered in the designing process by the software. After the fabrication of zirconia crowns, their primary seating on the metal dies was assessed. They were cleaned with distilled water in an ultrasonic bath for 5 minutes, and their external surface was polished with zirconia finishing points (DIACERA HP, EVE, Munich, Germany).

Surface treatment of specimens:

The crowns were then randomly assigned to 4 groups (N=10) for the following surface treatments:

Group 1 (control). This group served as the control group and did not receive any surface treatment.

Group 2 (AOP). The internal surface of crowns in this group was subjected to high-pressure argon-oxygen cold atmospheric plasma with 2% oxygen using atmospheric pressure plasma device (Plasma Research Technology Center, Mazandaran University, Babolsar, Iran) [6]. Plasma treatment conditions were 60 W, 7 kHz frequency of plasma reactor, and 15 kV peak to peak voltage. The optimal time (5 minutes for each specimen) and distance between the jet tip and specimen surface (5 mm) were determined according to the preliminary experiments measuring the contact angle [14].

Group 3 (AP). The internal surface of crowns in this group was subjected to 99.9% argon plasma with the same technique as explained for group 2.

Group 4 (SB). The internal surface of crowns in this group was subjected to sandblasting with 50

Table 1. Composition of materials used in this study

Material	Brand name, Manufacturer	Composition
AllCem Dual Dual cure cement	**FGM, Joinville, SC, Brazil**	Cement paste: Methacrylate monomers, camphorquinone, co-initiators, stabilizer, pigments, silanized barium, aluminium, silica glass microparticles, silicon dioxide nanoparticles, inorganic pigments, preservatives Catalyst paste: Methacrylate monomers, dibenzoyl peroxide and stabilizers, barium, aluminium, silica glass microparticles 67% of filler content
Ambar adhesive resin	**FGM, Joinville, SC, Brazil**	Urethan dimethacrylate, HEMA, methacrylate acidic monomers, methacrylate hydrophilic monomers, camphorquinone, silanated silicon dioxide, ethyl 4-dimethylaminobenzoate, ethanol
Panavia Sa **(self-adhesive, dual cure)**	**Kuraray, Noritake, Japan**	Paste A: Monomer (10-MDP, Bis-GMA, TEGDMA, HEMA, other methacrylate monomer), filler (silanated barium glass filler, silanated colloidal silica), initiator, pigment, others Paste B: Methacrylate monomer, filler (silanated barium glass filler, aluminium oxide, silanated sodium fluoride), accelerator, pigment, silane coupling agent, others
Filtek Z250	**3M ESPE, St. Paul, MN, USA**	Organic matrix: Trietilenglicol dimethacrylates (TEGDMA) < 1–5%; Bisphenol-A-glycidyl methacrylate (Bis-GMA) < 1–5%; Bisphenol-A polyethylene glycol diether dimethacrylate (Bis-EMA) 5–10%; Urethane dimethacrylate (UDMA) 5–10% Filler: Zirconia/silica
Zirconia blank	**Luxen Multi, Dental Max, Seoul, Korea**	ZrO2, HfO2, Y2O3, Y2O3, Al2O3, other oxides

μm Al2O3 particles (Cobra Renfert, Hilzingen, Germany) for 20 seconds in a sandblaster (FineBlast KFP Dental, Tehran, Iran) under 2 bar pressure such that the sandblaster tip had 10 mm distance from the specimen surface. The crowns were then cleaned in an ultrasonic bath (Ultrasound Vita-Sonic II Vita Zahnfabrik, Germany).

Cementation of zirconia crowns on metal dies:

The crowns were cemented on metal dies with Panavia SA self-adhesive resin cement (Kuraray Noritake, Okayama, Japan). Excess cement was removed from the margins by the sharp tip of an explorer after 2 seconds of light-curing and final light-curing was performed from the buccal and lingual surfaces for 20 seconds using a light-curing unit (Bluephase C8; Vivadent, Liechtenstein) with a light intensity of 800 mW/cm^2.

Prior to the FR test, metal dies were mounted in auto-polymerizing acrylic resin (Acropars, Tehran, Iran) and placed in a fatigue-testing machine (Nemo, Mashhad, Iran) to simulate masticatory forces. Each crown was subjected to 100 N load with 1 Hz frequency. The specimens were designed such that they could hold a stainless-steel ball with 3 mm diameter on their occlusal surface to ensure optimal load distribution. Totally, 1000 load cycles were applied to the crowns. Next, they were transferred to a universal testing machine (TB-5T, Koopa, Sari, Iran) to measure their FR. To simulate lateral masticatory forces, the specimens were mounted on the jig of a universal testing machine with 10-degree angulation. A stainless-steel ball with 3 mm diameter was placed at the center of crowns to ensure balanced load distribution. Next, compressive forces were applied to the specimens at a crosshead speed of 0.5mm/minute, and load at fracture was recorded (Figure 1).

Measurement of Microtensile bond strength:

Forty composite blocks measuring 3×5×5 mm were fabricated from Filtek Z250 composite resin (3M ESPE, St. Paul, MN, USA) with transparent plexiglass molds. Composite resin was manually applied as bulk in the mold, and a transparent celluloid tape and a glass slab were placed over it and compressed manually to create a smooth void-free surface. Light-curing was performed with a light intensity of 800 mW/cm^2 for 40 seconds. After removal of composite specimens from the mold, their bottom surface was also cured for another 40 seconds.

Forty zirconia blocks were cut into blocks measuring 3×6×6 mm by a precision sectioning machine (Delta, Mashhad, Iran), and polished with 400-, 600-, 800-grit silicon carbide abrasive papers under water coolant. They were then cleaned in distilled water in an ultrasonic bath for 10 minutes, and dried with air spray. The specimens were then sintered in a furnace (KFP dental auto sinter 1650; Kousha Fan Pars, Tehran, Iran) at 1500°C for 10 hours until they reached their final translucency and mechanical strength. Zirconia blocks were then assigned to 4 groups of 10, according to different surface treatments explained earlier. Next, each group was randomly assigned to two subgroups (N=5) based on the type of resin cement:

Subgroup 1: Z250 composite blocks were bonded to zirconia blocks using Panavia SA self-adhesive resin cement (Kuraray, Noritake, Japan) and cured from all four surfaces for 40 seconds.

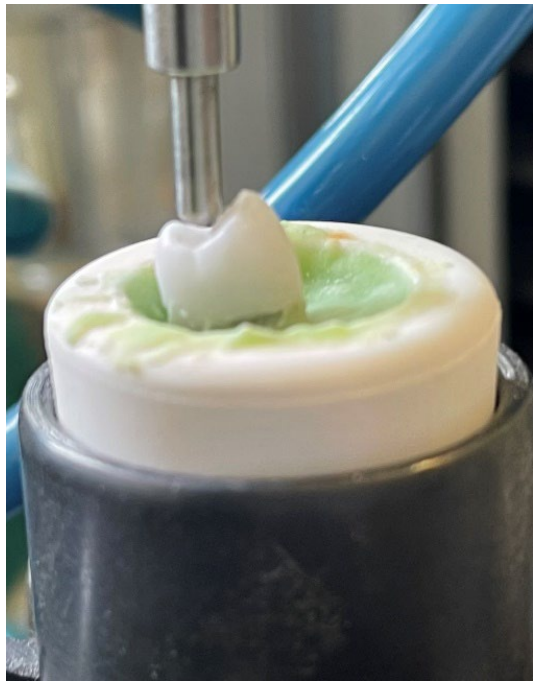

Fig. 1. Load application to a crown in a universal testing machine

Subgroup 2: Z250 composite blocks were bonded to zirconia blocks using Allcem Dual resin cement (FGM, Joinville, Brazil). For this purpose, composite blocks were etched with 37% phosphoric acid for 15 seconds, rinsed for 10 seconds, dried, and Ambar bonding agent (FGM, Joinville, Brazil) was applied and cured. Resin cement was then applied and cured from all four surfaces for 40 seconds.

The specimens were then incubated at 37°C for 24 hours to ensure their maximum polymerization, and subjected to 1000 thermal cycles between 5-55°C with a dwell time of 30 seconds. The specimens were sectioned by a precision sectioning machine, and each zirconia block cemented to composite was sectioned into 4 central rods measuring approximately 1 x 1 mm for MTBS test (N=20). MTBS was measured in a universal testing machine (TB-5T, Koopa, Sari, Iran) at a crosshead speed of 1 mm/minute. MTBS was calculated in megapascals (MPa) by dividing the load at fracture by the cross-sectional area of each rod measured by a digital caliper.

Mode of failure:

The mode of failure of specimens was inspected under a stereomicroscope (Dewinter technologies, Milano, Italy) at x40 magnification and categorized as: adhesive at the zirconia interface, adhesive at the composite interface, cohesive within the zirconia or composite resin, cohesive within the cement, and mixed including two or more of the above types (Table 2).

Assessment of SR by a profilometer:

Surface-treated zirconia blocks prepared for MTBS test (N=40), underwent SR assessment in a laser profilometer (Nemomechatronic; Mashhad, Iran) before being sectioned into rods, and the Ra value of each specimen was recorded with 1µm accuracy before and after surface treatment.

Assessment of surface topography:

One zirconia specimen from each group was selected and underwent atomic force microscopy (AFM) assessment and photographed for evaluation of surface topography and surface roughness. Two points from the periphery, two points at the center, and two points between the periphery and center were selected. Prior to scanning, all specimens were cleaned with cold air spray and alcohol. The resolution was 256 x 256, and the scan rate was 1 Hz.

Statistical analysis:

Data were analyzed using SPSS version 26 (IBM, Armonk, New York, USA). The normality of data distribution was assessed by the Shapiro-Wilk test, and homogeneity of the variances across the groups was analyzed by the Levene's test. The effect of surface treatment and cement type, and their interaction effect on MTBS were analyzed by two-way ANOVA. Differences among the groups in SR and FR were analyzed by one-way ANOVA and Tukey's post-hoc test. Independent t-test was applied to compare the MTBS values between the two resin cements. The level of statistical significance was set at 0.05.

RESULTS

Fracture resistance:

One-way ANOVA showed no significant difference in FR of the groups (P>0.05, Table 3).

Microtensile bond strength:

Two-way ANOVA showed that the effect of surface treatment on MTBS was significant (P<0.001), but the effect of cement type was not significant (P>0.05). Independent t-test

Table 2: Distribution of failure modes in the study groups (N=10)

Groups		Failure modes				
		I	II	III	IV	V
Argon plasma	AllCem	3	0	0	0	7
	Panavia SA	4	0	0	0	6
Argon-oxygen Plasma	AllCem	2	0	1	0	7
	Panavia SA	2	0	0	1	7
Sandblasting	AllCem	3	0	2	0	5
	Panavia SA	4	0	0	0	6
Control	AllCem	5	0	0	0	5
	Panavia SA	4	0	0	1	5

I: adhesive at the zirconia interface; II: adhesive at the composite interface; III: cohesive within the zirconia or composite resin; IV: cohesive within the cement; V: mixed including two or more of the above

showed no significant difference within each group in MTBS values between the two resin cements of Panavia SA and Allcem Dual (P>0.05). MTBS was significantly higher in SB and AOP groups than the control and AP groups for both Panavia and Allcem Dual cements (Table 4). Considering the mode of failure, adhesive (type 1) and mixed (type 4) failure modes had the highest frequency.

Surface roughness:

The SB group showed the highest SR, followed by the control, AOP, and AP groups. One-way ANOVA revealed a significant difference in the mean SR among the groups (P<0.001, Figure 2). Pairwise comparisons by the Tukey's test showed that the AP group had significantly lower SR than all other groups (P<0.05). Also, the SR of AOP group was significantly lower than that of control and SB groups (P<0.05). Figure 3 shows AFM images of zirconia surface subjected to different surface treatments.

DISCUSSION

This study assessed the effects of three different surface treatments on SR, FR, and MTBS of monolithic zirconia to resin cements. Regarding FR, no significant difference was found among the study groups. However, differences in SR were significant. Thus, the first hypothesis was partly rejected. The present results showed that SR in the AP group was significantly lower than that in the control group with no surface treatment. In line with the present findings, Tabari et al. [6] evaluated the effects of AP, AOP, their combination, and

Table 3. Mean FR (KN) of the study groups (N=10)

Group	Mean± SD	P
Control	1.85±0.49	0.948
Sandblasting	1.88± 0.46	
Argon-oxygen plasma	1.96±0.53	
Argon plasma	1.83±0.59	

SD: Standard deviation

Table 4. Means MTBS of zirconia to resin cements in the study groups (MPa)

	Control	Sandblasting	Argon-oxygen plasma	Argon plasma
Allcem Dual (FGM)	6.73±1.63[a]	9.91±1.74[b]	8.63±1.83[b]	7.22±1.36[a]
Panavia SA (Kuraray)	7.38±1.85[a]	10.45±1.88[b]	9.24±1.96[b]	7.64±1.35[a]
P	0.253	0.349	0.317	0.328

*Similar letters in each row indicate absence of a significant difference at 0.05 level

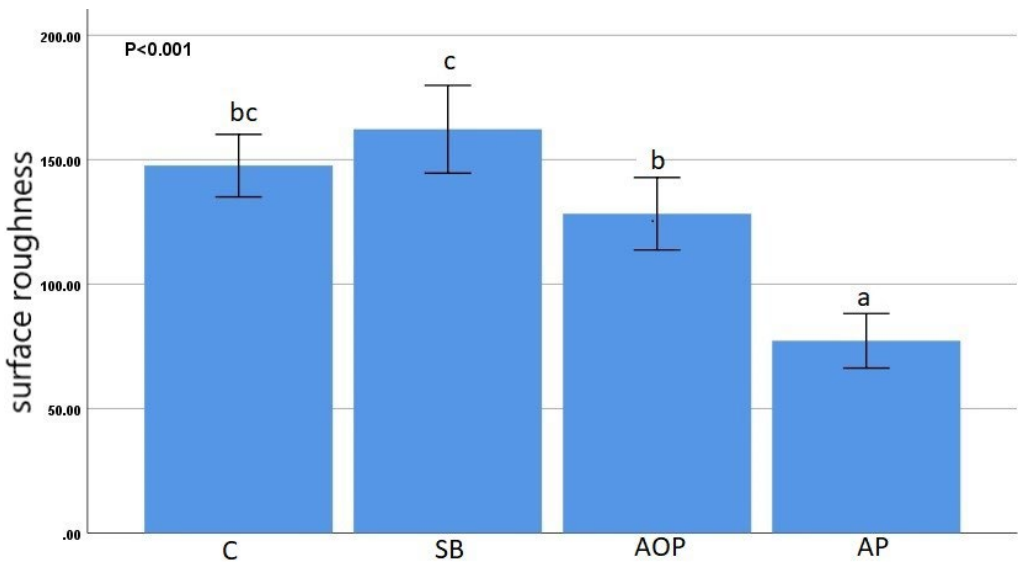

Fig. 2. Comparison of surface roughness (RA) of the study groups (N=10)
C: control; SB: sandblasting; AOP: Argon-oxygen plasma; AP: Argon plasma
*Similar letters indicate absence of a significant difference at 0.05 level

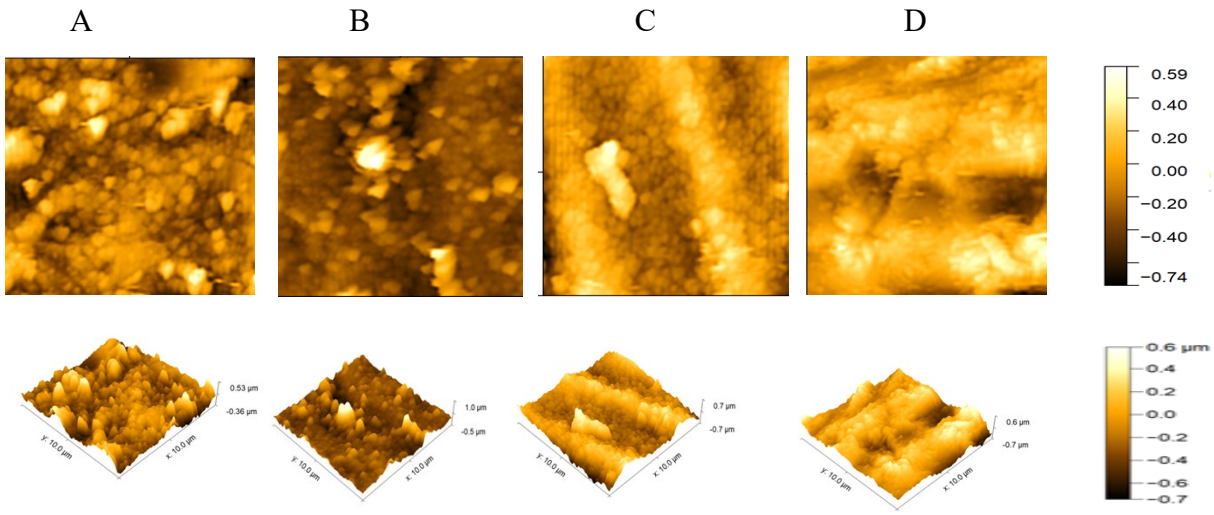

Fig. 3. Topographical evaluation of zirconia surface subjected to different surface treatments; (A) Argon plasma; (B) Argon-oxygen plasma; (C) control; (D) Sandblasting

sandblasting on surface properties of zirconia. They reported a significant reduction in SR following surface treatment with AP. Scanning electron microscopic images obtained after plasma treatment in a study by Vechiato-Filho et al, [15] revealed a change in the zirconia surface morphology, and replacement of the conventional matrix with a fine-grain matrix which can be a reason for decreased SR after AP treatment. Unlike the present results, Park et al. [16] evaluated the effect of AP at different time periods of 0, 24, 48, and 72 hours on SR and surface energy of zirconia crowns. They showed that application of AP decreased the SR compared with the control group, but not significantly, which can be due to differences in power of plasma device, duration of use, and distance from the specimen surface in the two studies.

In the present study, the increase in SR of translucent zirconia following sandblasting with 50 µm aluminum oxide particles was not significant compared with the control group. Consistent with the present results, Inokoshi et al. [17] reported that sandblasting with 50 µm aluminum oxide particles and 0.2 MPa pressure did not significantly change the SR of translucent zirconia. Moreover, Zhao et al. [7] showed that sandblasting of translucent zirconia with 50µm aluminum oxide particles and 0.1MPa pressure did not significantly change the SR compared with the control group. Unlike the present findings, Kim et al. [18] demonstrated that increasing the size of aluminum oxide particles from 25 µm to 110 µm significantly increased the SR of translucent and conventional zirconia.

In the present study, AOP significantly decreased the SR of translucent zirconia. This finding is contrary to the results of Tabari et al, [6] who showed that SR significantly increased with AOP. This controversy can be attributed to differences in oxygen percentage in AOP in the two studies since 2% oxygen was used in the present study while Tabari et al. [6] used 10% and 20% oxygen, and the SR increased with an increase in concentration of oxygen.

Regarding MTBS, the present results showed the highest MTBS values in SB and AOP groups, which were both significantly higher than the values obtained in other groups. The increased surface area of Y-TZP due to sandblasting enables more substantial surface reaction and greater contact among the hydrophilic monomers, cement, and zirconia ceramics. However, sandblasting also involves physico-chemical changes that can affect surface energy and wettability [7]. De Mendonça et al. [19] reported significant enhancement of bond strength by sandblasting, and showed that AP slightly increased the bond strength, which was not significant. Their results were similar to the present findings. Consistent with the present results, Tabari et al. [6] indicated that

application of AOP significantly increased the bond strength to zirconia, but the difference between AP and control groups was not significant.

Plasma cleans the surface by cleavage of C-H and C-C bonds and resultantly, eliminates the organic impurities and increases the surface energy as such [6]. Moreover, it has been reported that the plasma gas increases the formation of active peroxide radicals and other functional groups such as C-O and C-OH on the zirconia surface, which subsequently increase the surface energy [5]. It also decreases the contact angle between two surfaces and increases wettability. Thus, chemical attachments between the surface molecules improve, and the bond strength of zirconia to resin cement increases as such [20]. Higher MTBS in AOP compared with AP group can be attributed to increased oxygen at the surface of zirconia. Argon ions are inert; whereas, oxygen ions are reactive, and presence of oxygen ions on the surface subsequently increases the polarity of zirconia, which is a neutral agent. Valverde et al. [21] evaluated the effect of SB and AP on bond strength to zirconia. They showed that both SB and AP increased the bond strength. However, unlike the present results, they indicated that AP was more effective. This difference may be attributed to using a different type of zirconia in their study. Also, contrary to the present results, Negreiros et al. [22] reported an increase in bond strength to translucent zirconia following the application of AP. Difference between their results and the present findings may be due to measurement of bond strength after thermocycling in the present study. They measured the bond strength after 24 hours, and reported significantly higher bond strength in AP than the control group while they did not find a significant difference between the AP and control groups after 1 year of water storage.

The present results found no significant difference between Allcem Dual and Panavia SA resin cements in their bond strength to zirconia. However, the mean MTBS was slightly higher in the Panavia SA subgroup, which can be attributed to the presence of 10-MDP in its composition, and chemical interactions of the functional groups (phosphate and hydroxyl) in 10-MDP with the zirconia surface [7].

In the present study, no significant difference was found in FR of the study groups. Bozogullari et al. [23] evaluated the effect of zirconia surface treatment with SB and AP, and reported a significant increase in FR of conventional zirconia restorations following sandblasting. However, in Katana UTML cubic zirconia, type of surface treatment had no significant effect on FR, which was in agreement with the present results. The difference between cubic zirconia and the conventional 3Y-TZP zirconia is in lower rate of light reflection and higher translucency of the former type. Increased translucency is mainly due to mechanisms such as increased percentage of yttria that increases the cubic phase, which has lower light reflection than the tetragonal phase [7]. Also, phase transformation in zirconia depends on its microstructure, the amount of yttria, and the amount of cubic phase. Under compressive stresses, tetragonal-monoclinic phase transformation causes a volumetric expansion, prevents crack propagation, and can also increase the FR of restoration. The cubic crystalline phase, unlike the tetragonal crystalline phase, does not undergo phase transformation under stresses such as those caused by sandblasting, and this fact may explain absence of a significant difference in FR of translucent zirconia restorations in different groups in the present study. Negreiros et al. [24] found that surface treatment of zirconia with plasma did not induce phase transformation and had no adverse effect on its flexural strength. Furthermore, it has been demonstrated that zirconia surface treatment with plasma does not cause any destructive change in the zirconia surface [25].

Assessment of the mode of failure revealed no significant difference among the study groups, and adhesive and mixed failures had the highest frequency in all groups.

In vitro design was a limitation of this study. Another limitation was conduction of thermocycling before the sectioning of zirconia-composite blocks into rods, that may not reflect the clinical situation. Thus, generalization of results to the clinical setting

should be done with caution. Moreover, in the present study, bonding was performed immediately after plasma treatment; however, since the active surface state decreases with time, further research is required to assess the long-term effects of plasma surface treatment on durability of zirconia restorations.

CONCLUSION

The following conclusions were drawn:

1. AOP treatment improved the MTBS of resin cement to zirconia, and a bond strength comparable to that obtained by SB was achieved.
2. SR of translucent zirconia decreased following plasma treatment.
3- FR of zirconia crowns was not affected by SB or plasma treatment.

CONFLICT OF INTEREST STATEMENT

None declared

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