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Effects of different surface treatments of zirconia on the bond strength of self-adhesive resinous cement

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ABSTRACT

Purpose: The aim of this study was to evaluate the effects of different zirconia surface treatments on the bond strength of two self-adhesive resinous cements (SARC).
Methods: Two hundred and eight cylindrical specimens were obtained from Y-TZP zirconia (half with diameter 3.2 mm and half with 4.8 mm). After sintering and polishing, specimens were divided into four groups ($n = 26$), according to surface treatment: *Control* (no treatment); *Sandblasting* (Al_2O_3 particles); *Rocatec* (Al_2O_3 particles, tribochemical silica coating and silane application); *Laser* (Nd: YAG laser: 20 Hz, 100 mJ, 0.2 J/cm^2). The surface roughness (Ra) was evaluated after the surface treatments, and the groups were divided into two subgroups ($n = 13$), according to the SARC tested: RelyX U200 and Bifix SE. The 2.2-mm cylinders were bonded to 4.8-mm cylinders and stressed until failure under shear using a universal testing machine. Bond strength and Ra were analyzed using ANOVA, and Tukey's test ($\alpha = 0.05$).
Results: Surface treatment was significant ($p < 0.0001$), but cement type ($p = 0.73$) was not. Related to roughness, significant differences were found for the treatment type ($p < 0.0001$), with laser being the treatment with higher Ra values.
Conclusions: Nd:YAG laser produced a rougher surface and a higher bond strength compared with sandblasting, silicatization, and control groups.

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Zirconia; Nd:YAG Laser; surface treatments; bond strength; self-adhesive cement

Introduction

In recent years, the zirconia ceramic system has become popular because of its mechanical properties, such as high strength and toughness, as well as its great esthetic quality and chemical durability.[1,2] The clinical success of ceramic restorations depends on its mechanical properties and the cementation procedure, whereas a durable and stable bond between ceramics and luting agents is fundamental for the long-term performance of the indirect ceramic restorations.[3–5] However, the bond strength of zirconia to resin cements is not optimal yet,[3] as the zirconia presents a lack of the glass phase and the hydrofluoridric acid

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etching does not work on it.[6] Therefore, alternative methods for surface treatments of zirconia are necessary, and usually consist of achieving a chemical bond between zirconia and the resin cement, and improving the micromechanical retention.[7,8]

Among the alternative methods for zirconia surface treatment, airborne-particle abrasion (sandblasting), selective infiltration etching technique, plasma spraying, surface fluorination, tribochemical silica coating, Nd:YAG laser, Er:YAG laser, and CO_2 laser are listed in the literature.[3,9–14] Nonetheless, the surface conditioning methods of ceramics and category of luting cements have been proven to have great influence on the bond strength of all ceramic restorations.[15,16] Conditioning procedures in the surface of zirconia employing air abrasion with aluminum oxide particles or silicon oxide are usually employed in order to increase the adhesion between resin cements and prosthesis.[17–20] Liu et al. [3] reported that air abrasion with aluminum oxide improves the bonding properties of the zirconia, but excessive air abrasion can create microcracks, chipping, and loss of ceramic, compromising the mechanical properties of the material.[21]

Laser technology has been used in industrial surface treatment methods for more than 40 years. Nevertheless, only recently has the laser been used in dentistry for this purpose.[22] The use of the Nd:YAG laser has been suggested by some authors to improve the bond strength between cements and zirconia ceramics,[9,14,22,23] while others show that this improvement is lower when compared to silica coating and silane application.[13] Besides, it has been questionable whether the Nd:YAG laser can damage the zirconia surface, creating microstructural changes, crack formations, and consequently interfering in its mechanical strength.[24] There are few studies about the use of the Nd: YAG laser, and the ideal parameters of its application in ceramic surface treatments are controversial, hence, studies about this are still necessary.[22,25]

Therefore, the aim of this study is to compare the effects of the Nd:YAG laser over zirconia surface with conventional sandblasting and tribochemical silica coating on the bond strength of two self-adhesive resinous cements. The null hypothesis is that the use of the Nd:YAG laser for zirconia surface treatment does not increases the bond strength of the luting agents.

Materials and methods

Specimen preparation

Two hundred and eight zirconia cylinders with two different diameters (2.2 mm and 4.8 mm, $n = 104$ each) were obtained from Y-TZP zirconia blocks (e-max Zircad model B40 L, Ivoclair Vivadent, Liechtenstein) using a diamond trephine mill. After sintering, the cylinders were polished (DP-10, Panambra, Sao Paulo, SP, Brazil) with silicon-carbide abrasive papers (grit #1200, #2000, and #4000, Extec Corp, Enfield, CT, USA) for 90, 180, and 180 s, respectively, at 300 rpm. After each polishing, the specimens were immersed in an ultrasonic bath for 5 min (Ultrasonic Cleaner, Odontobras, Ribeirao Preto, SP, Brazil). The final height of the cylinders was 2 mm.

Specimens were divided into four groups ($n = 26$): *Control* (*Cont*) – no treatment; *Sandblasting* (*SB*) – with aluminum oxide particles (Rocatec Pre, 3 M/ESPE, St Paul, MN, USA) using an etching device with a distance of 10 mm between the zirconia surface and the device, inclination of 45°, and pressure of 2.5 bar for 10 s; *Silica coating* (*Si*) – sandblasting

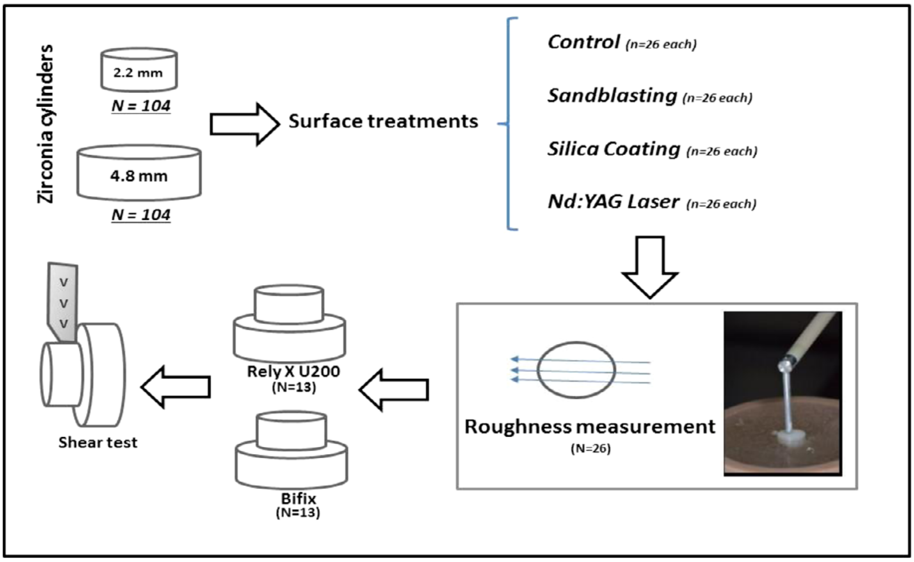

Figure 1. Chart of group division.

as described for the second group, followed by tribochemical silica coating with Rocatec Plus (3 M/ESPE) (110 μm) using an etching device with a distance of 10 mm between the zirconia surface and the device, inclination of 45°, and pressure of 2.5 bar for 10 s; and *Laser* (*L*) – application of the Nd:YAG laser (Pulse Master 600 IQ, American Dental Technologies, Inc., Corpus Christi, TX, USA) scanning the surface using an optical fiber of 320 μm in diameter, placed perpendicular and 1 mm distance from surface in a non-contact mode and no water spray; the zirconia surface was swept by the laser for 60 s. The laser parameters used were: 20 Hz with energy adjusted in 100 mJ.

After the treatments, all of the samples were cleaned using an ultrasonic bath for 20 min and air-dried. For the silica coating group, a silane agent (Ceramic Bond – VOCO GmbH; Cuxhaven, Germany) was applied over the treated surfaces for 1 min and then air-dried to remove the excess. Figure 1 shows the group divisions.

Zirconia surface roughness

The surface roughness of zirconia was evaluated measuring the mean surface roughness (Ra) of all the zirconia samples after the surface treatments and those of the control group. The Ra was obtained from profiles using a profilometer (MarSurf GD25, Mahr, Goettingen, Germany) coupled with dedicated measurement software (MarSurf XCR20, Mahr, Goettingen, Germany). The diamond stylus moved 2.5 mm along the sample; the first measurement starting 0.2 mm from the lower edge of specimen at a speed of 0.1 mm/s. Three measurements were performed for each specimen at intervals of 0.25 mm and a final average was obtained (Figure 1). The mean Ra values were determined with a cut-off value of 0.8 mm and a transverse length of 0.8 mm.

Table 1. Composition of all materials used in the study.

Material	Manufacturer	Batch Number	Composition
E-max Zircad,	Ivoclair Vivadent	K54200	Zirconium oxide (92%), yttrium oxide (5%), hafnium oxide (<2%), aluminum oxide + silicon oxide (<1%).
Bifix SE	VOCO	1,317,326	Bis-GMA, UDMA, Gly-DMA, phosphate monomers, initiators, stabilizers, glass fillers, aerosol silica (filler = 70 wt%).
RelyX U200	3 M/ESPE	488,822	Bis-GMA, TEGDMA, Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizer components, rheologic additives, alkaline fillers, pigments, rheologic additives (filler = 70 wt%).
Rocatec system	3 M/ESPE	419,361	Rocatec Pre: 110 µm aluminum oxide powder, Rocatec Plus: 30 µm silica-modified aluminum oxide coated with SiO_2
Ceramic Bond	VOCO	1,120,136	Silane (3-methacryloxypropyl trimethoxysilane), alcohol and water

Bonding procedure

From each group, two subgroups were created (n = 13) according to the self-adhesive resin cements tested: RelyX U200 (3 M/ESPE) and Bifix SE (VOCO GmbH, Cuxhaven, Germany). The composition of all the materials is listed in Table 1. The cements were manipulated following the manufacturer's instructions and placed over the treated surface of the smaller zirconia cylinders (diameter of 2.2 mm). Then, they were positioned over the larger cylinders (diameter of 4.8 mm) and a load of 100 g was applied over them. The excess was removed and, after 5 min, the cement was light-cured for 20 s on opposite sides using an LED light-curing unit with a power density of 600 mW/cm^2 (Emitter A, Schuster, Santa Maria, Rio Grande do Sul, Brazil). All samples were stored in deionized water for 1 week to allow for complete curing.

Shear test

The cylinder set was then stressed until failure under shear using a universal testing machine (DL-200MF, EMIC, São Jose dos Pinhais, PR, Brazil) with a knife-type punch, at a cross-head speed of 1 mm/min and load cell of 10 kgf, following the rules ISO/TS 11,405 (2003).

Scanning electron microscope analysis

Additionally, four small cylinders were obtained for scanning electron microscope (SEM) analysis. They received the same surface treatments (control, sandblasting, silica coating, and laser) and were sputtered with a gold alloy. The surface was observed by SEM (Inspect S50, FEI Company, Eindhoven, Netherlands), and the image from each surface was registered at X2,000 magnification, with the microscope operated at a voltage of 25 kV with a working distance of 14 mm.

Table 2. ANOVA results for Bond strength and Roughness (Ra).

ANOVA		p	SS	df	MS	F
Bond strength	Surface treatment	<0.0001	3087.86	3	1029.29	45.27
	Cement type	0.727	2.77	1	2.77	0.122
	Interaction	0.812	21.69	3	7.23	0.318
Ra	Surface treatment	<0.0001	58.85	3	19.61	572.06
	Cement type	0.724	0.004	1	0.0043	0.125
	Interaction	0.977	0.007	3	0.002	0.068

Table 3. Means (SD) of bond strength and results of Tukey's test for groups tested.

Treatment	Rely X U 200	Bifix
Control	7.28 (2.75) Aa	6.82 (2.77) Aa
Sandblasting	13.31 (3.92) Ba	12.03 (2.86) ABa
Rocatec	18.05 (3.89) Ca	17.32 (4.76) Ba
Laser	20.99 (8.17) Ca	22.16 (6.20) Ca

Note. Uppercase letters shows difference between columns (treatment) and lowercase letters shows difference between lines (cement type).

Statistical analysis

The bond strength and roughness data were checked for normality assumption using Kolmogorov–Smirnov test with 5% significance level. The shear bond strength data ($n = 13$) were analyzed using a two-way ANOVA (Surface treatment × Cement type), followed by Tukey's test ($p < 0.05$). The Ra data ($n = 26$) were analyzed using a one-way ANOVA, and also followed by Tukey's test ($p < 0.05$). The correlation between the shear bond strength and the roughness data was analyzed using Pearson's correlation.

Results

The results of a two-way ANOVA (Table 2) for bond strength evaluation showed significant differences only for the surface treatment groups, which revealed significantly higher values for silica coating group and laser group. For the cement type and the interaction between them, no differences were found, thus Bifix SE and RelyX U200 had a similar performance regarding the bond strength values. The means of bond strength for the treatments and the results of Tukey's test for surface treatment are seen in Table 3.

In relation to roughness, the results of a one-way ANOVA (Table 2) showed significant differences only for the surface treatment, with the laser group presenting the higher values, followed by the sandblasting and silica coating groups. The control group presented the lowest roughness values. The means and the results of Tukey's test are seen in Table 4.

Despite the Nd:YAG laser treatment producing an increase in the surface roughness and presenting a higher bond strength compared with all of the groups, the Pearson correlation between Ra and shear bond strength was only moderate ($r = 0.63$), and is shown in Figure 2.

The SEM analysis of the surface's morphology from specimens revealed the presence of cracks in the zirconia treated with the laser (arrows in Figure 3(D–F)), with an appearance of melting. The sample from the group submitted to sandblasting (Figure 3(B)) shows a very porous surface, with the same aspect as the one from the silica coating group (Figure 3(C)). Figure 3(A) shows an example of the sample from the control group with a very smooth and flat surface. The scratches presented in Figure 3(A) originated from the polishing procedure.

Table 4. Means (SD) of Ra and results of Tukey's test for treatments.

Groups	Mean ± SD (Ra)
Control	0.06 (±0.04) a
Sandblasting	0.33 (±0.04) b
Silica coating (Rocatec)	0.40 (±0.04) b
Nd:YAG Laser	1.98 (±0.35) c

Note. The groups followed by the same letters did not present significant differences.

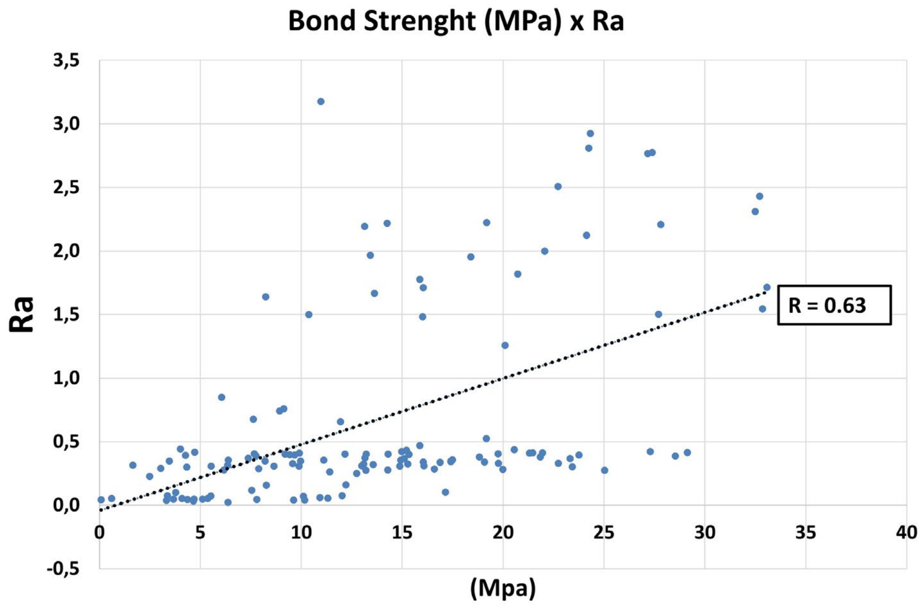

Figure 2. Results of the correlation test between bond strength and roughness.

Discussion

According to the present findings, the null hypothesis was denied, because the zirconia bond strength was affected by the use of the Nd:YAG laser. The application of the Nd:YAG laser increased the zirconia surface roughness and improved the bonding performance of both cements tested.

The results of the present study showed that the SB group presented higher bond strength values only when compared with the Cont group with a high polished surface, in which no treatment was performed. This treatment creates a rough surface that increases the surface area, formed due to the high-speed impact of aluminum particles. Indeed, Figure 3(B) shows the SEM image from one specimen treated with sandblasting, evidence of the roughness created on the surface by the aluminum particles. However, although the sandblasting created a rough surface, this treatment *per se* cannot create enough roughness to provide a reliable bonding with the resin cement.[26,27]

The treatment employed in the Si group (sandblasting followed by silica coating) has also been reported as an effective method to increase the bond strength of resin cements to zirconia.[14] Besides creating a rough surface, the silica coating allows the chemical interaction between the silica-modified zirconia surface and the silane coupling agents.[12] The

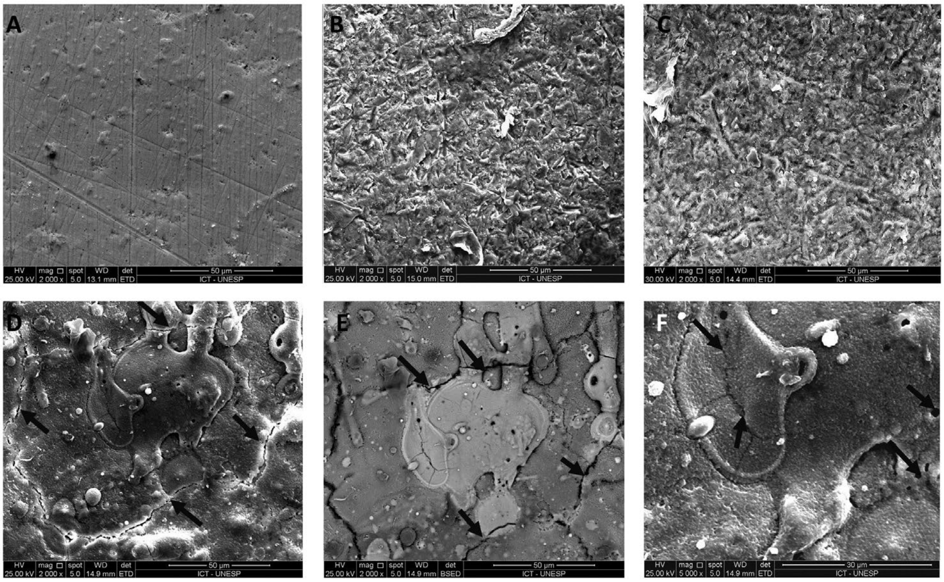

Figure 3. SEM images from the zirconia's surface in the four different groups tested: (A) Control; (B) Sandblasting; (C) Silica coating; and (D) Nd: YAG laser. (E) Same picture shown in D, detected with Back-Scattered Electron Detector evidencing the microcracks (arrows) in the zirconia surface. (F) Nd: YAG laser treatment evidencing holes and cracks (arrows). Images A to D and F were taken with Secondary Electron Detector (ETD). Images A to E are presented with X2000 augmentation, and image F with X5000.

group treated with Rocatec Plus (Si group) presented a higher bonding performance than the SB group, even without a significant difference in roughness between them, indicating that the silica coated on the zirconia surface could have been responsible for enhancing the bonding. In Figure 3(C), the SEM image from a specimen in the Si group shows a similar aspect with a specimen from the SB group, also indicating that the bond strength increase in the silica coating group might have been caused by the silica layer and silane interaction.

Although sandblasting and silica coating treatments had improved the bond strength of the resin cements tested, the group treated with the Nd:YAG laser presented the higher bonding performance of all the groups tested. Indeed, previous studies showed that the use of this laser increases the bond strength values of Y-TZP zirconia to resin cement compared with the use of sandblasting and silica coating,[9,23,28] due to an increase in the zirconia surface roughness. The Nd:YAG laser acts over zirconia by removing content from its surface due to the punctate action of laser-induced microexplosions (ablation), resulting in the formation of voids. Additionally, it also promotes fusing and melting of the most superficial ceramic layer followed by solidification to a smooth, blister-like surface.[29] SEM analysis shows that the sample sandblasted (Figure 3(B)) and the one with silica coating (Figure 3(C)) present a visually rougher surface than the sample irradiated with laser (Figure 3(D)). The laser sample presents some kind of melting pattern that induced a better bond strength than the other treatments for Bifix SE and RelyX U200 (except for silica coating group, which was similar). Also, this melted surface after laser irradiation created a Ra mean value almost five times higher than the other treatments, contradicting the visual parameters shown by SEM (Figure 3). A possible explanation is that the Ra measured in this study is the most common parameter used and uses the differences in height from peaks and valleys of the surface. The melting pattern created by the laser might have promoted higher peaks and valleys that increased the Ra values, while the sandblasting and silica coating treatments promoted higher number of these peaks. This explains the moderate correlation between roughness and bond strength ($r = 0.63$) found in this study. In addition, the energy and laser parameters used in most studies are not standardized, varying from 100 to 200 mJ,[14,22,30,31] which makes the comparison between studies difficult. The energy used in this study was 100 mJ which promoted some cracks in the zirconia surface (Figure 3(D–F)). Although these cracks might favor the penetration of resin cement and improves the bond strength, their effects on zirconia's mechanical properties should be better investigated.

In relation to the adhesive cements, some studies recommend the use of cements containing the phosphate ester agent 10- methacryloyloxydecyl dihydrogenphosphate (MDP),[14,32] since the functional phosphate ester group of MDP combines directly with metal oxides, and the combination of alumina air abrasion and MDP has been expected to generate stable bonding between a Y-TZP ceramic surface and resin-based adhesives.[33] However, the type of surface conditioning of zirconia seems to interfere in the bonding performance more than the type of cement used.[34] Although the resin cements used in this study did not present the MDP monomer in their formula, both are indicated for zirconia bonding, acting by micromechanical retention with the zirconia surface.[35] RelyX U200 is an improved version of RelyX U100, but there are few *in vitro* and clinical studies about it, especially regarding zirconia bonding. The bonding performance of the RelyX U100 was reported to be satisfactory in Y-TPZ zirconia crowns, with success rates around 97% after 5 years,[23] and the same must be expected for the RelyX U200. The different self-adhesive resin cements used, RelyX U200 and Bifix SE, presented a similar bond and could be an alternative for zirconia bonding. An increase in the bond strength presented by both cements tested in the sandblasting, silica coating, and laser groups is probably due to an increase in the roughness compared to the control group, allowing a better retention between the cements and the zirconia surface.

This study was conducted *in vitro*, evaluating the bond strength of the zirconia–zirconia interface. Thus, different results can occur in the zirconia–dentin interface, and must be tested. Also, additional studies must be done to find new methods to improve the bonding properties of zirconia, in order to verify the best laser parameters for the Nd:YAG and other lasers, like Er:YAG and CO_2. Additionally, *in vivo* studies can also be conducted to verify the longevity of zirconia bonding to tooth structures and the retention rates of crowns cemented with self-adhesive cements.

Conclusion

Despite the moderate correlation between roughness and shear bond strength, the use of the Nd:YAG laser improved the bonding performance of both resin cements tested, being similar to silica coating for RelyX U200. The influence of cracks formed after the laser application in the mechanical properties of zirconia must be further studied. Regarding the resin cements tested, RelyX U200 performed similarly to Bifix SE.

Disclosure statement

No potential conflict of interest was reported by the authors.

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