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The Effect of Femtosecond Laser Treatment on the Effectiveness of Resin-Zirconia Adhesive: An In Vitro Study

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Abstract
Introduction: When aesthetics is compromised, dental ceramics are excellent materials for dental restorations; owing to their optical properties and biocompatibility, zirconia ceramics are particularly interesting. Self-adhesive resin cements are the most suitable for bonding to zirconia ceramics, but traditional adhesive chemistry is ineffective and surface treatments are required to improve the adhesive bonding between resin and zirconia. The aim of this study was to evaluate the effect of femtosecond laser treatment on the shear bond strength (SBS) of self-adhesive resin cement on zirconia surfaces and to contrast it with other different surface conditioning methods.
Methods: Sixty square-shaped zirconia samples were divided randomly into four groups (n=15) according to their surface conditioning method: the NT group - no surface treatment; the APA25 group-airborne abrasion with 25 µm alumina particles; the TSC group-tribochemical silica coating, and the FS group-femtosecond laser irradiation (800 nm, 4 mJ, 40 fs/pulse, 1 kHz). Self-adhesive resin cements were bonded at the centre of samples, and after 72 hours, they were tested for SBS with a universal testing machine at a crosshead speed of 0.5 mm/min, until fracture. Five zirconia surfaces for each group were subjected to a surface morphology analysis by scanning electron microscopy (SEM). The failure modes were noted and a third of the specimens were prepared to morphological analysis.
Results: The NT group showed lower SBS values than the other groups. Femtosecond laser treatment demonstrated higher values than the control and APA25 groups and similar values to those of the TSC group. In the APA25 group, the surface conditioning method had values close to those of the TSC group, but lower than those obtained with femtosecond laser treatment.
Conclusion: The treatment of zirconia with femtosecond laser irradiation created a consistent and profound surface roughness, improving the adhesive effectiveness of the zirconia-resin interface.
Keywords: Femtosecond laser; Zirconia; Shear bond strength; Adhesion; Surface treatment.

Introduction
Currently, aesthetic dentistry has become one of the main areas of interest in dentistry. When aesthetics is compromised, dental ceramics are excellent materials for dental restorations; specifically, zirconia ceramics offer an excellent option.[1,2] They have several important characteristics, such as good optical properties and biocompatibility, and achieve good adhesion values that, clinically, minimize debonded surfaces.[3] Zirconia ceramics are currently the focus of clinical and industrial activity and extensive research.[4,5]

Zirconia ceramics display the fundamental characteristics and properties of biomedical appliances, especially when the zirconia is partially stabilized with yttrium oxide (Y_2O_3).[6,7] Yttria tetragonal zirconia polycrystal (Y-TZP) ceramics exhibit increased hardness, high strength, fracture toughness, wear resistance, low thermal conductivity, good frictional and non-magnetic behavior, and corrosion resistance to acids and alkalis, among others properties.[8,9] Regarding the bonding to Y-TZP, among the resin cement luting systems that we use for bonding, self-adhesive resin cements are the most adequate due to their properties and the simplicity of the cementation technique.[10-12]

Traditional adhesive chemistry is not useful on zirconia

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surfaces[13] and surface treatments are required to increase the adhesive bonding with the resin cements.[14-16] The most popular clinical surface conditioning techniques to improve the resin-zirconia interface are sandblasting,[17-19] silica coating,[20-22] carbon dioxide (CO_2) laser irradiation, and Erbium-Doped Yttrium Aluminum Garnet (Er:YAG).[23,24] Nevertheless, there is still no ideal zirconia surface conditioning method able to provide sufficient effectiveness of adhesion on zirconia surfaces. It is therefore necessary to find an adhesion protocol with a view to obtaining a resin-zirconia bond with stability and persistence.[25,26]

One of the new surface treatments currently being explored is irradiation with femtosecond laser, which produces optical pulses lasting femtoseconds (1 fs = 10^{-15} s). Within the group of femtosecond lasers, is the Titanium: Sapphire laser, which is based on a femtosecond Ti: Sapphire centenary oscillator producing wavelengths near the infrared (795 nm) and energies in the range of 10 nanoJ, with a repeat rate of 80 MHz. This system has an output amplifier, creating a pulse of 120 fs.

It would be of great clinical use if femtosecond lasers could be employed to perform the conditioning of biological and non-biological surfaces on which elements are cemented in Dentistry. Some studies have already addressed the enamel-bracket interface,[27,28] obtaining encouraging results in the sense of improvements in adhesive effectiveness and the possibility of replacing traditional conditioning agents that cause biological damage to tooth enamel. Regarding adhesion to porcelain, in a recent study, the authors analyzed zirconia surface conditioning with a femtosecond laser to improve the porcelain-bracket interface.[29] It was reported that femtosecond lasers could represent a new alternative to conventional surface treatments, and they have therefore become a new study object in the search for improvements in the interface adhesion of this non-biological surface; in this sense they could become a valid option for improving resin-zirconia interface adhesion, as proposed here.

The aim of this study was to evaluate the effect of femtosecond laser treatment on the shear bond strength (SBS) of self-adhesive resin cements to zirconia surfaces and to compare such treatment with more conventional surface conditioning methods.

Methods

The study used 60 square-like samples, which measured 6 × 6 × 1 mm, of sinterized Y-TZP (Cercon®, DeguDent, Hanau, Germany). All the specimens were wet-polished with 600-grit silicon carbide paper and assigned blindly into four experimental groups commensurate with the surface conditioning method to be performed (n = 15):

Group 1: Control- No treatment (NT): No surface treatment was applied.

Group 2: Airborne particle abrasion with 25 μm alumina particles (APA25): The zirconia surface was sandblasted with 25 μm alumina particles (Al_2O_3) that were applied at a perpendicular distance of 10 mm with a pressure of 0.25 MPa and duration of 20 seconds.

Group 3: Tribochemical silica coating (TSC): The zirconia surface was treated with the Rocatec system (Rocatec™ Soft, 3M Espe, Seefeld, Germany) using 30 μm alumina with silica particles. Silica coating was applied with the same parameters as the previous group. After that, specimens were silanized with a Rely X™ ceramic primer (3M Espe, Seefeld, Germany).

Group 4: Femtosecond laser (FS): The zirconia surface was irradiated with a commercial Ti:sapphire oscillator-regenerative amplifier system (Mantis-Legend, Coherent) that generated 1 kHz trains of pulses centered at 800 nm, during 40 fs, with an energy of 4.0 mJ which was precisely controlled with a variable neutral-density filter and quantified with a thermal detector (S302C, Thorlabs). The samples were attached on a computer-controlled XYZ-motorized stage (PT3-Z8, Thorlabs) and the laser pulses were focused on the zirconia surface with a fused silica lens (f = 100 mm), producing a spot diameter of 20 μm. Thus, the pulse energy was 0.015 mJ, the scanning velocity 0.25 mm/s and the scanning step 0.02 mm.

Once all surface conditioning methods were performed, a self-adhesive resin cement was bonded on the zirconia samples: Clearfil™ SA Cement (CLE) (Clearfil™ SA Cement, Kuraray, Osaka, Japan) using a cylindrical silicone mould with 3 mm internal diameter, 1 mm thickness and 1 mm in height.

The mould was placed in the centre of zirconia surfaces and the cement was applied over them, and then polymerized for 40 seconds (XL 3000, 3M/ESPE; light intensity 500 mW/cm^2, distance 0) parallel and perpendicular to the contact area. Once the mould was removed, the resin cement cylinder bonded to the zirconia surface, was light cured for 40 seconds.

All zirconia samples, with the cement cylinder on their surface, were kept in distilled water at 37°C for 72 hours and tested for SBS with a universal testing machine (AGS-X Autograph, Shimadzu Corporation, Kyoto, Japan), applying a shear load with a crosshead speed of 0.5 mm/min, until fracture. The bond strength values (in MPa) were obtained by dividing the maximum load registered until failure (in Newtons) by the bonding area (in millimetres).

All fractured samples were analyzed under an Axio M1 (Carl Zeiss, Germany) light microscope at ×40 magnification to register the failure modes. These were divided in adhesive (no remnants of resin cement on the zirconia surface, failure in adhesion) or mixed (zirconia samples showing residual cement on their surface, with both adhesive and cohesive failures).

Five samples from each surface method conditioning were subjected to surface morphology analysis with a variable pressure scanning electron microscopy (SEM) (Zeiss EVO MA25; Carl Zeiss, Jena, Germany) at different magnifications (×70 and ×1000), to determinate the effect of laser irradiation and the other surface treatments.

Additionally, several fractured samples were dehydrated for 48 hours in a desiccator (Sample Dry Keeper Simulate Corp., Tokyo, Japan) and were sputter-coated with a 10-nm platinum layer in a SEM coating unit (Polaron Equipment Ltd., Hertfordshire, England, UK) to analyze, with the scanning electron microscope at different magnifications (from ×30 to ×1000), the morphology of the debonded interfaces and to find differences among the surface topography of the surface treatments.

Statistical Analysis

The bond strength values (in MPa) were analyzed with SPSS version 21 (SPPS Inc., Chicago, IL), with $P<0.05$ as the threshold for statistical significance. Analysis of variance (ANOVA) test was employed to evaluate the differences obtained in the SBS test between the zirconia surface treatment groups. Bonferroni post hoc comparisons were used to quantify these inter-groups differences.

In addition, the chi-square test was applied to find differences in failures modes among the groups. The results were recorded in a table of 2 × 2. The control group was employed as reference to determinate the risk of adhesive failure (odds ratio [OR] and relative risk).

Results

The values obtained in SBS test (the mean values and standard deviations [SD]) for all the groups are shown in Table 1.

The results recorded with ANOVA revealed that the variance of SBS within the groups was significantly different ($P<0.001$). The Bonferroni post hoc inter-group comparisons indicated that the control group (NT) had lower SBS values than the other groups. Femtosecond laser treatment offered higher values than the control and APA25 groups and similar values to those seen for TSC. The surface conditioning method in the APA25 group had values close to those of the TSC group, but lower than those obtained with femtosecond laser treatment.

Bond failure-modes analyses are shown in Table 2. In the control group, the majority of failures were adhesive, while in other treatment groups they were mostly mixed. The 2 × 2 failure rate comparison using the reference control group revealed significant differences in the TSC and FS groups ($P=0.06$); but not in the APA25 group ($P=0.143$). The risk of adhesive failure was significantly higher in the control group than in the TSC and FS (OR = 5.5) groups.

Figure 1 shows the SEM images of zirconia surface morphology after all the surface conditioning methods had been employed. Figures 1A and 1B represent the NT group at a magnification power of 70 and 1000 respectively. They show marked scratches running in the same direction as result of the polishing procedure. Figures 1C and 1D that correspond to the APA25 group, display granule-shaped micro-retentions over the surface, due to the impact of the high-speed 25 µm alumina particles. Figures 1E and 1F, which represent the TSC group, show a rough appearance as a result of the coating and solidification of the tribochemical silica particles. The FS group (Figures 1G and 1H) show a well-defined pattern of deep horizontal furrows.

Figure 2 represents several SEM images of debonded zirconia surfaces, after SBS, at two different powers of magnification. Figure 2A shows adhesive failure with no visible remains of the resin cement. Figures 2C, 2E and 2G display mixed failures with residual cement on the zirconia surface with a large visible cohesive phase. Details of the debonded areas can be observed in Figures 2B, 2D, 2F, 2H at a magnification power of 700.

Discussion

Y-TZP ceramics are now very popular dental restoration materials owing to their exceptional properties, such as fracture toughness and biocompatibility.[30] However, their long-term success depend not only on these properties, but also on others, such as the type of cement,[31] the cementing procedure[32] and, especially, the surface rough-

Table 1. Means and SD of the SBS Values (MPa) Obtained in the Experimental Groups

Zirconia Treatment Surface	No Treatment	Airborne Particle Abrasion	Tribochemical Silica Coating	Femtosecond Laser Irradiation
SBS	Mean 4.4^{A}, SD 1.3	Mean 8.1^{B}, SD 3.6	Mean 9.5^{BC}, SD 2.3	Mean 10.8^{C}, SD 1.9

Abbreviation: SBS, shear bond strength.
Different letters refer to significant inter-group comparisons after Bonferroni corrections.
$F=19.4$; $P<0.001$.

Table 2. Modes of Bond Failure and Risk of Adhesive Failures Taking the Control Group as Reference

	NT Group (n = 15)	APA25 Group (n = 15)	TSC Group (n = 15)	FS Group (n = 15)
	No. (%)	No. (%)	No. (%)	No. (%)
Adhesive	10 (66.7)	5 (33.3)	4 (26.7)	4 (26.7)
Mixed	5 (33.3)	10 (66.7)	11 (73.3)	11 (73.3)
Relative risk (95% CI)	2.3 (1.0-5.1)	0.5 (0.2-1.1)	0.4 (0.2-1.0)	0.4 (0.2-1.0)
OR (95% CI)		4.0 (0.9-18.3)	5.5 (1.2-26.4)	5.5 (1.2-26.4)

Abbreviations: OR, odds ratio; TSC, tribochemical silica coating; FS, femtosecond; NT, no treatment, APA25, airborne abrasion with 25 µm alumina particles.

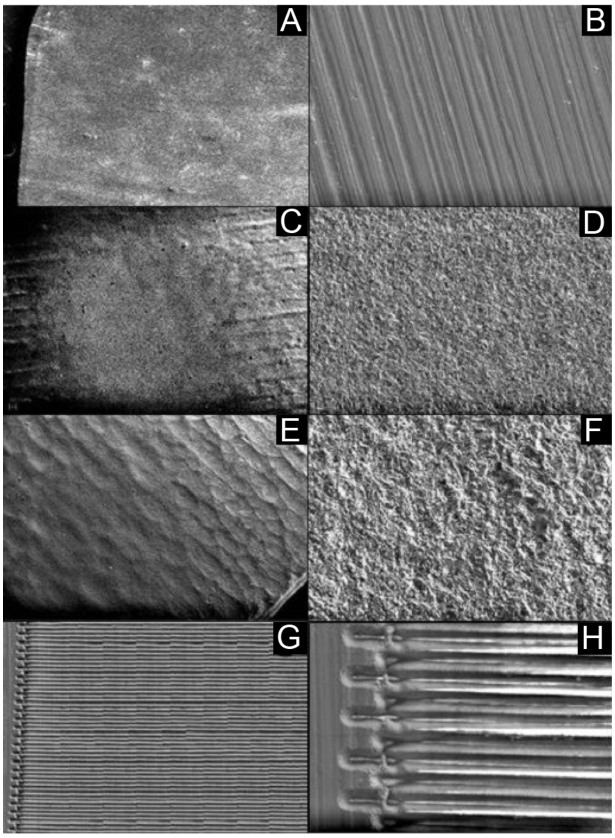

Figure 1. Scanning Electron Microscopy (SEM) Micrographs of Zirconia Ceramic Surfaces After Conditioning Treatments (x70 and x1000 magnification). A, B: No treatment (NT); C, D: Airborne-particle abrasion with 25-µm alumina particles (APA25); E, F: Tribochemical silica coating (TSC); G, H: Femtosecond laser (FS).

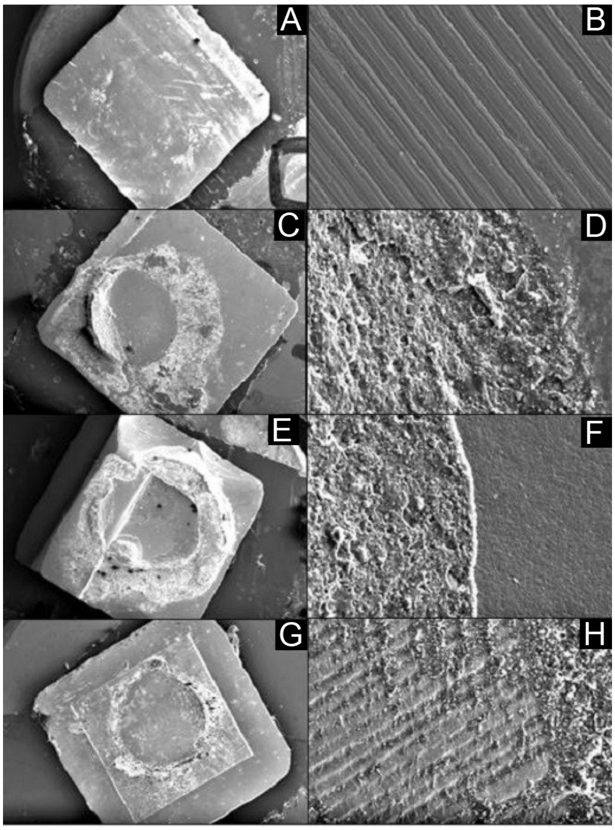

Figure 2. Representative Zirconia Surface Scanning Electron Microscopy (SEM) Images (x30 and x700 magnification) of the Most Common Failure Type for Each Group. A and B (NT group) shows an adhesive failure where there are no visible resin cement remains on the ceramic surface due to complete detachment; C and D (APA25 group); E and F (TSC group); G and H (FS group) show mixed failure, with a cohesive phase and visible pores in the remaining residual cement.

ness, because the surface conditioning procedure is an important element in bonding to zirconia surfaces.[15, 25]

The bond strength values evidenced that femtosecond laser irradiation was more competent in improving bond strength than airborne-particle abrasion with 25-µm alumina particles and no surface treatment (Table 1). The SEM observations showed substantial quality changes in the zirconia surface topography after femtosecond laser irradiation (Figure 1G) as compared with the other surface treatments, and this could be related to higher values (10.8 MPa). Kara et al[24] have demonstrated that femtosecond laser irradiation is an efficient surface conditioning method for roughening the surfaces of zirconia ceramics. It produces a pattern of deep horizontal furrows on the zirconia surface (Figure 1H), allowing greater retention of resin cements, which become intertwined within these grooves to form a single bonded structure, thereby increasing bond strength. In contrast, in the control group (Figure 1B) there was no abrasion or irregularities on the surface that could provide micromechanical retention, so it only showed the effect of wet-polished with 600-grit silicon carbide paper. Also, in the APA25 group, alumina particle sandblasting created a rough surface (Figure 1D), but the surface irregularities had insufficient micro depth and therefore both groups exerted less mechanical retention than the FS group (Figure 1H) and TSC group (Figure 1F).

In recent studies[27,28] an abrasion pattern similar to that obtained in our study when applying femtosecond laser on the enamel surface, also showing increased adhesive efficiency, has been reported. The use of a femtosecond laser may be valid for conditioning enamel and zirconia surfaces, as we showed here. Thus, the irradiation with femtosecond lasers is increasingly supported as an alternative in the field of adhesion in dentistry, replacing drills in conservative dentistry[33] and also serving as both a conditioning agent and for the ablation of different types of surfaces -whether they are biological, such as the enamel[27,28] or dentine,[34] or non-biological, such as porcelain[29] or zirconia, as in the present case- with a minimum amount of thermal and mechanical damage to the surfaces.[35,36]

The TSC group had SBS values close to those of the FS group. This high value (9.5 MPa) can be directly related to the architecture of the zirconia surface after application of the tribochemical silica coating (Figure 1E), which mainly affects the bond strength results because the surface roughness seems to be a more relevant factor in bonding to zirconia surfaces.[33]

In view of the SBS values obtained, although the pattern of engraving is different than that achieved with the femtosecond laser, because the FS group (Figure 1H) has a horizontal stripe pattern and the TSC group (Figure 1F) creates irregularities that do not obey any specific pattern, both are equally retentive. It may therefore be concluded that the retention depends more on the engraving depths obtained than on the type. Accordingly, besides the influence of the roughness of the surface created, resin cement adhesion could be effectively improved by silica coating on zirconia surfaces.[37] In addition, silane enables the chemical adhesion.[22] After the silica particles have impacted the surface, the zirconia surface irregularities are infiltrated by ceramic primer Rely X™. Silane coupling agents produce superior contact and infiltration of the resin into the zirconia surface irregularities and protect against moisture through chemical bonding. In their study May et al demonstrated that the application of the MDP-primer increments bond strength.[20]

Airborne-particle abrasion with 25-μm Al_2O_3 particles was less efficient in improving bond strength than femtosecond laser irradiation. Although alumina particle sandblasting created a rough surface (Figure 1C) similar to that seen in the TSC group (Figure 1E) it does not enhance bond strength because the surface irregularities have insufficient micro depth (Figure 1D), unlike the irregularities created in the FS (Figure 1H) and TSC (Figure 1F) groups, and therefore fails to generate sufficient micromechanical retention. In a recent study, Akpinar et al tested the zirconia-bracket interface with the use of a cementing agent. Although our study focused directly on the zirconia-resin interface, disregarding differences the data can be said to be comparable.[29]

The Control group showed lower SBS values than the other groups. This can be seen from the SEM images (Figure 1A), where it may be observed that in these zirconia samples, which were only subjected to wet-polishing with 600-grit silicon carbide paper, there were no surface irregularities (Figure 1B) such as shallow pits or micro-cracks to provide micromechanical retention. The Y-TZP zirconia surface roughness is influenced by the surface treatment applied and is directly related to the bonding properties.[38] Aboushei et al demonstrated that a strong and durable resin-zirconia bonding is vital for the longevity of dental restorations and this is only possible with a surface treatment that will allow the zirconia surface to be roughened.[25]

Specific zirconia surface areas were analyzed by SEM to compare the topography and morphology of the debonded interfaces of each group. The bond failure modes assessed are shown in Table 2. They support the bond strength results and the differences among the experimental groups. The Control group showed a tendency towards adhesive failure at the resin-zirconia interface, without residual resin cement on the zirconia surface (Figure 2A), in accordance with the literature addressing resin cements.[39] In the APA25, TSC and FS groups mainly mixed failures were observed (Figure 2B, 2C, 2D) with significant differences in the TSC ($P = 0.06$) and FS groups, but not in the APA25 group ($P = 0.143$).

The predominance of mixed failures and the fact that the adhesive failure occurred primarily in the Control group showed that the different bond strength values among the experimental groups were related with the differences in surface roughness between the zirconia samples treated with the surface conditioning methods (Figure 1).

Conclusion

Zirconia treatment with femtosecond laser irradiation and tribochemical silica coating creates consistent roughness on its surface, improving the adhesive effectiveness of the zirconia-resin cement interface, with higher early bond strength values in the FS group.

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Ethical Considerations

None to be declared.

Conflict of Interests

The authors declare that they have no conflict of interest.

References

1. Denry I, Kelly JR. State of the art of zirconia for dental applications. *Dent Mat.* 2008;24:299-307. doi:10.1016/j.dental.2007.05.007.
2. Vagkopoulou T, Koutayas S, Koidis P, Strub JR. Zirconia in Dentistry: Part 1. Discovering the nature of an upcoming bioceramic. *Eur J Esthet Dent*. 2009;4:130-51.
3. Manicone PF, Iommetti PR, Raffaelli L. An overview of zirconia ceramics: basic properties and clinical applications. *J Dent.* 2007;35:819-826. doi:10.1016/j.jdent.2007.07.008.
4. Gomes AL, Montero J. Zirconia implant abutments: a review. *Med Oral Pato Oral Cir Bucal.* 2011;16:50-55. doi:10.4317/medoral.16.e50.
5. Ozcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. *J Adhes Dent.* 2015;17:7-26.
6. Calvalti AN, Foxton RM, Watson TF, Oliveira MT, Giannini M, Marchi GM. Y-TZP ceramics: key concepts for clinical application. *Oper Dent*. 2009;34:344-351. doi:10.2341/08-79.
7. Nakonieczny D, Walke W, Majewska J, Paszenda Z. Characterization of magnesia-doped yttria-stabilized zirconia powders for dental technology applications. *Acta Bioeng Biomech.* 2014;16:99-106.
8. Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. Strength and reliability of surface treated Y-TZP dental ceramics. *J Biomed Mater Res*. 2000;53:304-313.
9. El-Korashy DI, El-Refai DA. Mechanical properties and

bondig potencial of partially stabilized zirconia treated with different chemomechanical treatments. *J Adhes Dent.* 2014;16:365-376.
10. Ferracane JL, Stansburry JW, Burke FJT. Self-adhesive resin cements–chemistry, properties and clinical considerations. *J Oral Rehabil*. 2011;38:295-314. doi:10.1111/j.1365-2842.2010.02148.x.
11. Casucci A, Goracci C, Chieffi N, et al. Microtensile bond strength evaluation of self-adhesive resin cement to zirconia ceramic after different pre-treatments. *Am J Dent*. 2012;25:269-275.
12. Maeda FA, Bello-Silva MS, de Paula Eduardo C, Miranda Junior WG, Cesar PF. Association of different primers and resin cements for adhesive bonding to zirconia ceramics. *J Adhes Dent*. 2014;16:261-265.
13. Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. *Dent Mater.* 1998;14:64-71. doi:10.1016/s0109-5641(98)00011-6.
14. Elsaka SE. Effect of surface treatments on the bonding strength of self-adhesive resin cements to zirconia ceramics. *Quintessence Int*. 2013;44:407.
15. Usumez A, Hamdemirci N, Koroglu BY, Simsek I, Parlar O, Sari T. Bond strength of resin cement to zirconia ceramic with different surface treatments. *Lasers Med Sci.* 2013;28:259-566. doi:10.1007/s10103-012-1136-x.
16. Moradabadi A, Roudsari SE, Yekta BE, Rahbar N. Effects of surface treatment on bond strength between dental resin agent and zirconia ceramic. *Mater Sci Eng C Mater Biol Appl*. 2014;34:311-317. doi:10.1016/j.msec.2013.09.015.
17. Akin H, Ozkurt Z, Kımalı O, Kazazoglu E, Ozdemir A. Shear bond strength of resin cement to zirconia ceramic after aluminium oxide sandblasting and various laser treatments. *Photomed Laser Surg*. 2011;29:797-802. doi:10.1089/pho.2011.3039.
18. Gomes AL, Castillo-Oyagüe R, Lynch CD, Montero J, Albaladejo A. Influence of sandblasting granulometry and resin cement composition on microtensile bond strength to zirconia ceramic for dental prosthetic frameworks. *Am J Dent*. 2013;4:31-41. doi:10.1016/j.jdent.2012.09.013.
19. Sarmento HR, Campos F, Sousa RS, et al. Influence of air-particle deposition protocols on the surface topography and adhesion of resin cement to zirconia. *Acta Odontol Scand.* 2014;72:346-353. doi:10.3109/00016357.2013.837958.
20. May LG, Passos SP, Capelli DB, Ozcan M, Bottino MA, Valandro LF. Effect of silica coating combined to a MDP-based primer on the resin bond to Y-TZP ceramic. *J Biomed Mater Res B Appl Biomater*. 2010;95:69-74. doi:10.1002/jbm.b.31684.
21. Queiroz JR, Massi M, Nogueira L Jr, Sobrinho AS, Bottino MA, Ozcan M. Silica-based nano-coating on zirconia surfaces using reactive magnetron sputtering: effect on chemical adhesion of resin cements. *J Adhes Dent.* 2013;15:151-159.
22. Gomes AL, Ramos JC, Santos del Riego S, Montero J, Albaladejo A. Thermocycling effect on microshear bond strength to zirconia ceramic using Er.YAG and tribochemical silica coating as surface conditioning. *Laser Med Sci.* 2015;30:787-795. doi:10.1007/s10103-013-1433-z.
23. Kasraei S, Rezaei-Soufi L, Heidari B, Vafaee F. Bond strength of resin cement to CO2 and Er:YAG laser-treated zirconia ceramic. *Restor Dent Endod.* 2014;39:296-302. doi:10.5395/rde.2014.39.4.296.
24. Kara O, Kara HB, Tobi ES, Ozturk AN, Kilic HS. Effect of various lasers on the bond stregth of two zirconia ceramics. *Photomed Laser Surg.* 2015;33:69-76. doi:10.1089/pho.2014.3841.
25. Aboushei MN. Evaluation of zirconia/resin bond strength and interface quality using a new technique. *J Adhes Dent.* 2011;13:255-260.
26. Cvikl B, Dragic M, Franz A, Raabe M, Gruber R, Moritz A. Long-term storage affects adhesion between titanium and zirconia using resin cements. *J Adhes Dent*. 2014;16:459-464.
27. Lorenzo MC, Portillo M, Moreno P, et al. In vitro analysis of femtosecond laser as an alternative to acid etching for achieving suitable bond strength of brackets to human enamel. *Lasers Med Sci.* 2014;29:897-905. doi:10.1007/s10103-013-1278-5.
28. Lorenzo MC, Portillo M, Moreno P, et al. Ultrashort pulsed laser conditioning of human enamel: in vitro study of the influence of geometrical processing parameters on shear bond strength of orthodontic brackets. *Laser Med Sci.* 2015;30:891-900. doi:10.1007/s10103-013-1491-2.
29. Akpinar YZ, Irgin C, Yavuz T, Aslan MA, Kilic HS, Usumez A. Effect of femtosecond laser treatment on the shear bond strength of a metal bracket to prepared porcelain surface. *Photomed Laser Surg*. 2015;33:206-212.
30. Camposilvan E, Marro FG, Mestra A, Anglada M. Enhanced reliability of yttria-stabilized zirconia for dental applications. *Acta Biomater.* 2015;17:36-46.
31. Passos SP, May LG, Barca DC, Ozcan M, Bottino MA, Valandro LF. Adhesive quality of self-adhesive and conventional adhesive resin cement to Y-TZP ceramic before and after aging conditions. *Oper Dent.* 2010;35:689-696. doi:10.2341/10-157-l.
32. Inokoshi M, de Munck J, Minakuchi S, Van Meerbeek B. Meta-analysis of bonding effectiveness to zirconia ceramics. *J Dent Res*. 2014;93:329-334. doi:10.1177/0022034514524228.
33. Luengo MC, Portillo M, Sánchez JM, et al. Evaluation of micromorphological changes in tooth enamel after mechanical and ultrafast laser preparation of surface cavities. *Lasers Med Sci*. 2013;28:267-273. doi:10.1007/s10103-012-1144-x.
34. Portillo M, Luengo MC, Sánchez JM, et al. Morphological alterations in dentine after mechanical treatment and ultrashort pulse laser irradiation. *Lasers Med Sci.* 2012;27:53-58. doi:10.1007/s10103-010-0845-2.
35. Pike P, Parigger C, Splinter R, Lockhart P. Temperature distribution in dental tissue after interaction with femtosecond laser pulses. *Appl Opt*. 2007;46:8374-8378. doi:10.1364/ao.46.008374.
36. Girard B, Cloutier M, Wilson DJ, Clokie CMI, Miller RJD, Wilson BC. Microtomographic analysis of healing of femtosecond laser bone calvaria wounds compared to mechanical instruments in mice with and without application of BMP-7. *Lasers Surg Med*. 2007;39:458-467.
37. Liu D, Tsou JK, Matinlinna JP, Wong HM. Effects of some chemical surface modifications on resin zirconia adhesión. J *Mech Behay Biomed Mater.* 2015;46:23-30.
38. Kirmali O, Kustarci A, Kapdan A, Er K. Efficacy of surface roughness and bond strength of Y-TZP zirconia after various pre-treatments. *Photomed Laser Surg.* 2015;33:15-21. doi:10.1089/pho.2014.3825.
39. De Oyagüe RC, Moticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Influence of surface treatments and resin cement selection on bonding to densely-sintered zirconium-oxide ceramic. *Dent Mater*. 2009; 25:172-179. doi:10.1016/j.dental.2008.05.012.