Effect of airborne-particle abrasion and, acid and alkaline treatments on shear bond strength of dental zirconia

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The surface roughness, morphology and shear bond strength (SBS) of dental zirconia using three different surface treatment techniques were evaluated. Three groups of sintered zirconia blocks were treated as follow, 1) Airborne-particle abrasion (APA) group (G1-APA), 50- μm Al_2O_3 ; 2) APA and 9% hydrofluoric acid etching (G2-HF); 3) APA and Sodium Hydroxide (G3-NaOH). The specimens were evaluated for roughness [atomic force microscope (AFM)], morphology [Scanning Electron Microscope (SEM)] and for SBS in the universal testing machine. The AFM revealed changes in the roughness after the surface treatments, however there was not R_a difference between groups, SEM analysis revealed changes in surface morphology for all surface treated specimens. For SBS, significant difference was found between G1-APA=8.4 $\pm$ 2.7 MPa and G2-HF=3.3 $\pm$ 0.6 MPa ($p<0.05$) and G2-HF and G3-NaOH=9.0 $\pm$ 3.0 MPa ($p<0.05$). The main fracture mode was mixed failure (63%) for G1-APA and G3-NaOH groups. G2-HF showed 100% adhesive failure. SBS was improved with NaOH, however application of HF significantly decreased SBS.

Keywords: Zirconia, Adhesion, Surface treatment, Alkaline treatment, Acid treatment

INTRODUCTION

Due to non-silica based structure of zirconia, conventional techniques for adhesion of porcelains are not useful for this non silicate ceramics¹. Therefore, the first attempts to improve resin and zirconia adhesion were directed in creating a silica cover on the surface of zirconia and then using the traditional adhesive system of silanation². Silica coating of zirconia has been attempted through, silica tribochemical coating, silicon nitride hydrolysis³, glazing porcelain on full sintered zirconia, use of silica slurry before sintering⁴, *in situ* silica nanoparticle surface deposition⁵, and glass bead cover before sintering⁶. Airborne-particle abrasion (APA) with Al_2O_3 and silica-coated aluminum of different granulometry are used as mechanical treatment⁷⁻¹⁰. Chemical surface treatment includes acid treatments, hydroxylation and monomers use. As hydrofluoric (HF) acid is not effective on zirconia degradation at conventional clinical concentrations at room temperature, different concentrations, temperature and/or exposure time have been tested, namely 40% concentration for 210 s¹¹, 48% concentration at 100°C for 25 min¹², 9.5% concentration at 25°C for 1, 2, 3 or 24 h or at 80°C for 1, 3, 5 or 30 min, and 48% HF concentration at 25°C for 30 to 60 min¹³. Another chemical approach is through surface treatment using monomers. It has been reported the effectiveness

of luting agents containing a hydrophobic phosphate monomer such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP)¹⁴ after that report, the aim was to determine whether or not it was consistent, leading to formulate, hypothesis regarding a chemical adhesion. In recent years chemical bonding evidence between zirconia surface and MDP monomer has been reported, because the presence of a chemical group of phosphate monomer (P)-O-Zr identified by time-of-flight secondary ion mass spectrometry after MDP surface treatment on zirconia¹⁵, while other research explained the chemical coupling mechanism between MDP and zirconia surface using a computational model, which showed that the coupling mechanism occurs in double-coordinate and single-coordinate configurations and that in the coupling process there are the possibility of an acid environment. Furthermore, they concluded that application of MDP in alkaline conditions improves shear bond strength (SBS)¹⁶. Regarding pH it has been reported that the use of MDP-based primers and alkaline solution contributed in increasing zirconia bond strength¹⁷. Hydroxylation of bioinert zirconia surface was also proposed to increase the -OH groups so as to improve wettability and the reaction between surface and MDP resin cement monomers¹⁸. However, few studies had been conducted regarding zirconia surface treatment using different pH solutions, for what the objective of this research was to evaluate the effect of three different surface treatments, namely mechanical and acid and alkaline solutions,

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on dental zirconia SBS, since the dual cured luting agents to adhere zirconia could be affected by the pH environment. The null hypothesis states that there are not SBS differences between three surface treatments to adhere zirconia.

MATERIALS AND METHODS

The brand, batch number and the composition of the materials used in this study are summarized in Table 1.

Specimen preparation

One-hundred fourteen zirconia square blocks, (12.5×12.5×5 mm) were cut from five presintered commercial dental zirconia discs (Zenostar, Wieland Dental+Technik, Pforzheim, Germany) in a CAD-CAM system (Zenotec Mini, Wieland Dental+Technik) using 1.0 mm carbide drills. Once manufactured, the blocks were sintered in a furnace (Zenotec Fire Cube, Wieland Dental+Technik) in standard set following the manufacturer instructions. The expected contraction was approximately 25%, and the final zirconia block size was 10×10×4 mm. The zirconia blocks were individually mounted in an autopolymerizing acrylic resin (Nic Tone, MDC Dental, Guadalajara, Mexico) in a custom-made tray with square-shaped molds (2×2×1 cm). Polymerizing was done while immersed in water to counter the exothermic effect. Ninety composite cylinders (Clearfil AP-X Esthetics, Kuraray Noritake Dental, Tokyo Japan), with a 5-mm diameter and 2-mm thickness, were made in a nonstick matrix. Then, each side of the cylinder

was light-cured with Bluephase N MC 800 mW/cm² (Ivoclar-Vivadent, Schaan, Liechtenstein) for 20 s as per manufacturer instructions —at 1 mm distance without touching the composite surface according to the light-curing guideline¹⁹. Before adhesion process, composite cylinders were ultrasonically cleaned in 96% ethanol for 2 min and were dried at room temperature (22–23±2°C). Zirconia samples were randomly assigned in three groups; 1) APA (G1-APA), 2) APA and HF acid (G2-HF), 3) APA and sodium hydroxide (G3-NaOH, gp/n=36) wherein gp/n=30 were for bond strength test, gp/n=5 for roughness measurement using atomic force microscope (AFM) and gp/n=1 for morphologic examination using scanning electron microscope (SEM). For the SBS, power value for the sample size in this study was calculated as *post hoc* using the highest and lowest mean value found for SBS and their respective standard deviations, and $\alpha=0.05$, on the basis of these values the power was >0.80.

Surface treatment and adhesion procedure

Zirconia blocks were treated by use of APA in a blaster (Eco Basic, Renfert, Hilzingen, Germany) with 50- μ m Al₂O₃ (Zeta Sand, Zhermack, RO, Italy) at a 90° angle 10-mm distance, 1 bar of pressure, 30 seg/cm², and with a 1-mm diameter blaster tip. To standardize the procedure, a custom-made appliance was used to keep the distance, angle, and free movement across the samples, which were ultrasonically cleaned in 96% ethanol for 2 min and were dried with gentle oil-free air for 10 s. For G1-APA, only APA treatment was applied.

Table 1 Comoposition, batch number, and manufacturers of materials used in the present study

Material	Composition	Batch no.	Manufacturer
Zeta Sand	99.80% Al ₂ O ₃	U112682/A	Zhermack Technical
Zenostar MT	Zirconium oxide (ZrO ₂ +HfO ₂ +Y ₂ O ₃)≥99.0%, Yttrium oxide (Y ₂ O ₃)>6.5≤8.0% Hafnium oxide (HfO ₂)≤5%, Aluminium oxide≤1.0%	U35562	Wieland Dental
Panavia F 2.0	A paste:10-methacryloyloxydecyl dihydrogen phosphate (MDP), dl-Camphoroquinone, catalyst and initiators; paste B: hydrophobic aromatic dimethacrylate, barium glass treated sodium fluoride, catalyst, accelerators and pigments	7D0107 7D0010	Kuraray Noritake
ED Primer II	Liquid A: 2-hydroxyethyl methacrylate (HEMA), MDP monomer, B: N-Methacryloyl-5-aminosalicylic acid, accelerators and water.	7A0024 7A0144102023	—
Clearfil Ceramic Primer	3-Methacryloxypropyl trimethoxy silane, MDP monomer and ethanol	720011	Kuraray Noritake
Clearfil AP-X Esthetics	Bisphenol-A-diglycidyl methacrylate (Bis-GMA) Hydrophobic aromatic dimethacrylate, dl-Camphorquinone, silanated barium glass filler and pre-polymerized organic filler	5CD143	Kuraray Noritake
Porcelain Etch	9% Hydrofluoric Acid	BCG4P	Ultradent
Silane	Silane	BBY3G	Ultradent
Sodium Hydroxide	NaOH 99%	144102	Fermont

For G2-HF, after APA the surface was treated with 9% HF acid, (Porcelain Etch, Ultradent Products, South Jordan, UT, USA) for 2 min, the HF was removed with water spray and was then ultrasonically cleaned as mentioned above. Before adhesion, a generous quantity of silane was applied in the surface and was left to dry for 2 min (Silane, Ultradent Products). For G3-NaOH, after the APA procedure, a 0.01-M NaOH solution (Sodium Hydroxide, CAS-1310-73-2, Fermont, Monterrey, Mexico) with a pH of 13.5 was generously applied and was left to dry for 10 min prior to adhesion as previously reported¹⁶. For all groups, a ceramic primer containing MDP-10 was applied generously (Clearfil Ceramic Primer, Kuraray Noritake Dental) for 5 s and left to dry for 40 s. Composite cylinders were also conditioned as per manufacturer instruction. Dual curing cement (Panavia F 2.0, Kuraray Noritake Dental) was mixed and applied at zirconia surface. The composite cylinder was positioned on the zirconia surface using cotton pliers, and afterwards, a light finger pressure was applied for 10 s. The excess cement was removed with a microbrush, and the oxygen-blocking agent was applied (Oxyguard, Kuraray Noritake Dental). After 5 min, it was removed with gentle water spray. All the specimens were prepared by the same operator at room temperature ($22\text{--}23\pm 2^\circ\text{C}$). The samples were stored in deionized water ($37\pm 2^\circ\text{C}$) immediately after completion of the bonding procedure for 24 h and before SBS testing.

Surface roughness and morphology

After the surface treatment, five randomly selected samples from each experimental group and one control group ($G0/n=5$) were evaluated through AFM (NaiAFM, Nanosurf, Liestal Switzerland). A $50\times 50\text{ }\mu\text{m}$ area was measured in tapping mode to determine the average roughness (R_a). Six measurements were taken from each sample, thus giving a total of 30 measurements per group. For morphologic examination, the representative samples in each group and one control sample ($n=1$) were observed with an SEM (JEOL, JSM-6510LV, Tokyo, Japan) and after gold sputtering, the examination was conducted at 20-kV acceleration and $2,000\times$ magnification. For surface roughness the data were analyzed using a statistical program (SPSS 22.0 IBM, Chicago, IL, USA). Thereafter, descriptive statistics was obtained, the results were tested for normal distribution using the Shapiro-Wilk method. Kruskal-Wallis test, was carried out, and U-Mann-Whitney, as pairwise *post hoc* analysis, was performed after the data failed the test for normal distribution.

SBS test and fracture mode

The samples were immediately tested for SBS after being removed from water, in a universal testing machine (Autograph AGS-X, Shimadzu, Kyoto Japan), and great caution was enforced so as not to apply any force to the adhesive area during mounting in the apparatus. The test was performed at 1 mm/min crosshead speed, and the debonding load was registered in MPa. After SBS test, all specimens were examined in a stereoscopic

microscope at $40\times$ magnification by a single observer to determine the failure mode, which was classified with the modified adhesive remnant index (ARI) criteria in which: Score 0=No adhesive left on zirconia bonding surface (adhesive failure); Score 1=Less than half of the adhesive is left on zirconia bonding surface; Score 2=More than half of the adhesive is left on zirconia bonding surface; and Score 3=All adhesive is left on zirconia bonding surface²⁰. Mixed failure for 1–2 scores and cohesive failure for score 3. Thereafter, descriptive statistics was obtained, the Shapiro-Wilk, Kruskal-Wallis test, and U-Mann-Whitney was applied for SBS. Moreover, statistical differences were investigated for ARI score by Chi-square test.

RESULTS

Roughness values (R_a) measured with AFM (in μm) and group comparison are shown in Fig. 1. Mean values per group are as follows: $G0=0.20\pm 0.07\text{ }\mu\text{m}$, $G1\text{-APA}=0.29\pm 0.01\text{ }\mu\text{m}$, $G2\text{-HF}=0.27\pm 0.09\text{ }\mu\text{m}$, and $G3\text{-NaOH}=0.28\pm 0.11\text{ }\mu\text{m}$. The values of experimental groups are comparable, with APA having the highest mean value. Kruskal-Wallis test revealed that the surface treatment had significant effect on surface roughness ($p<0.05$). U-Mann-Whitney test revealed significant differences between control group and all experimental groups ($p<0.05$). However, no significant difference was observed between APA group and chemical treatment groups $G1\text{-APA}$; $G2\text{-HF}$ ($p=0.779$); $G1\text{-APA}$; $G3\text{-NaOH}$ ($p=0.824$); and $G2\text{-HF}$; $G3\text{-NaOH}$ ($p=0.842$), which suggests that the chemical treatment has no significant effect on surface roughness.

The surface morphology micrographs are presented in Fig. 2, AFM surface micrographs show peaks and valleys that can be observed in experimental groups, whereas the crests and valleys observed in control group

Fig. 1 Roughness comparison between groups; G0: control; G1-APA: APA; G2-HF: APA/9% HF and G3-NaOH: APA/NaOH through AFM. U-Mann-Whitney's test ($p<0.05$) different letters are significantly different groups.

Fig. 2 Surface morphology after surface treatment, SEM×2,000 magnification, 10 µm bar (left) and AFM (right) micrographs.

A1–A2: control; B1–B2: APA; C1–C2: APA/Hydrofluoric Acid; D1–D2: APA/Sodium Hydroxide.

are lower and more homogeneous. SEM micrographs exhibited similar morphology for experimental groups; however, G1-APA showed more surface irregularities than the other groups. GC exhibited a regular surface, and neither scratches nor cracks were observed in any group. AFM and SEM micrographs are congruent with R_a values.

SBS values and the comparison between groups are shown in Table 2. The highest mean value belongs to the G3-NaOH surface treatment, followed by G1-APA and the lowest mean value belongs to G2-HF. Nonparametric test Kruskal-Wallis revealed the effect of surface treatments on SBS of zirconia ($p<0.05$) and U-Mann-Whitney test showed differences between experimental groups.

The distribution frequency and percentages score for failure mode after debonding are shown in Table 3. The Chi-Square comparison for ARI scores between groups indicated that the groups were not all the same (Chi-square=22.46, $p<0.001$). The predominant for G1-APA and G3-NaOH reveals mixed failure. For G2-HF treatment, all the samples have an adhesive failure. The score of 3 was not found for any group, Table 4.

Table 2 Mean and standard deviation (SD) for SBS (MPa) after 24 h water storage and Kruskal-Wallis analysis

Surface treatment	SBS
G1-APA	8.4 ^A (2.7)
G2-HF	3.3 ^B (0.6)
G3-NaOH	9.0 ^A (3.0)

Mean values represented with different superscript uppercase letters are significantly different according to U-Mann-Whitney's test ($p<0.05$)

Table 3 Distribution, frequency and percentage of modified ARI scores

Surface treatment	0 (%)	1 (%)	2 (%)	Total
G1-APA	4 (13.3)	19 (63.3)	7 (23.3)	30
G2-HF	30 (100)	0 (0)	0 (0)	30
G3-NaOH	9 (30)	19 (63.3)	2 (6.6)	30
Total	43 (47.7)	38 (42.2)	9 (10)	90

Score 0: Adhesive failure; 1 y 2: Mixed failure. Chi-square/Fisher's test ($p<0.001$)

Table 4 Failure mode by surface treatment group

Group	Adhesive	Cohesive	Mixed
G1-APA	4	0	26
G2-HF	30	0	0
G3-NaOH	9	0	21
Total	43	0	47

DISCUSSION

The main objective of this study was to investigate the effect of three different surface treatments on SBS of dental zirconia resin cement interface, based on pH differences in each technique, and the null hypothesis states that there are not SBS differences between three surface treatments to adhere zirconia. The null hypothesis was rejected in this study, since there were found significant differences between groups.

Even several techniques for zirconia adhesion has been investigated, after a meta-analysis report that focused on zirconia adhesion, the authors stated that data reporting on zirconia treatments requires more standardization as it is of great importance to scientists gathering evidences regarding this matter²¹. Most of reported studies *in vitro* used slow-cut blade instruments followed by polishing zirconia surface before the adhesion procedure^{3,6,8-10}. The common process of zirconia restorations in clinical practice is through computer aided design-computer aided manufacture (CAD-CAM), and the zirconia specimens used in this study were manufactured using the mentioned technology, which let us have a real approach regarding the roughness effect of the surface treatments in zirconia, as we measured the roughness on zirconia surface as it has been manufactured using CAD/CAM and after undergo to surface treatment. An important predicting variable to improve the SBS is the surface roughness. In previous studies some authors concluded that the APA of zirconia surface before the bonding procedure could be a key in success regardless of the particle granulometry¹⁰. On the other hand, it has been published that there is a significant difference in surface roughness with respect to distance, and that there is significant difference as well in adhesive strength when the zirconia surface is treated by APA in different angles regardless of the distance⁸. In this study, all experimental groups were treated by APA, and the findings of the previous researches, which specified the respective angles and distances for APA, and the instructions of the manufacturer regarding pressure and granulometry were all taken into account. In our research, the zirconia surface roughness of the control group, after it was designed and cut using the CAD/CAM system, was reported, which could be a very important fact since the clinicians are getting zirconia inside surface restorations in similar conditions, so that the surface modification after surface treatment in this research was carried out close to the real dental practice. This study shows the roughness parameter R_a through AFM measurement after different surface treatments namely combined mechanical and chemical treatment with an acid solution (HF 9%) and an alkaline solution (NaOH 0.01M, pH 13.5). Statistical analysis revealed that all experimental groups are significantly different with respect the control group, which is in line with the results from previous studies wherein researchers found that all APA specimens have shown a higher surface roughness than the control group after APA of the zirconia surface^{3,4,8}.

On the other hand, no difference was found between the experimental groups, and this can be due to the same mechanical surface treatment for all of them. Additional chemical treatments used in the study appear to have no effect on surface zirconia crystals degradation under the used protocol, and it consequently have no effect on surface roughness. There are some evidences that 9.5% HF acid was able to modify the zirconia surface. However, the exposition time is at least 1 h immersion at 25°C or 1 min immersion at 80°C¹³. In a similar study that is in line with our findings, for sodium hydroxide solution, no morphologic differences were found after the comparison of SEM micrographs of APA and acid and alkaline surface treatment¹⁶. In this study observations of the surface with SEM showed that additional chemical treatment with acid or alkaline solution after APA did not alter substantially the surface morphology of all experimental groups, which is consistent with the computed R_a values. As roughness is an important factor that promotes the mechanical interlocking between adhesive luting agent and zirconia surface, the nonexistence of difference between groups can be a good indicator that our results with respect to SBS are satisfactory. Is roughness a determinant of SBS or is it the chemical treatment in our study? For SBS, the lower value was computed for acid solution surface treatment, which have significant difference with those of APA and alkaline solution groups. The bond strength is greatly influenced by 50 μm sized APA and in combination with MDP based product SBS achieved the highest mean, due to the APA treatment enhance the wettability and the MDP promotes the bond strength²². Then if we have the same surface interlocking possibilities, and the chemical bonding by the MDP containing adhesive and resin cement we could suggest that under the same surface conditions in all groups, the results on SBS could be attributed to the effect of the chemical treatments acid and alkaline instead of the mechanical treatment. It can also be concluded that HF treatment have a negative effect on SBS under the conditions of our study, and these findings agree with a similar research in which the lowest mean value on their study was computed for the acid pH treatment group as compared with the neutral treatment group and alkaline surface treatment group¹⁶.

This can be attributed to formed P-O-Zr complex as the adhesive MDP-10 reacts with the zirconia surface¹⁵, H^+ ion dissociation is necessary to give place to the P-O-Zr complex; and as a consequence, it has been suggested that high levels of H^+ could weaken the bond strength¹⁶. The pH of adhesive systems influence bond strength of chemically cured resin materials, thus resulting in lower bond strength value for lower pH monomers, and this can be explained by the existing competition between residual acidic monomers and benzoyl peroxide for tertiary amine²³. In the other hand, even some evidences of chemical bonding between MDP monomer and zirconia surface has been reported^{15,16}, the mechanism is not fully explained. There are reported three possible mechanisms of MDP interaction with zirconia; hydrogen

bonding, ionic bonding and both mechanisms in the same phosphate monomer²⁴), the authors propose that the MDP-zirconia interaction could occur in a neutral pH environment, as result only one P-OH group can interact with either zirconia or neighboring phosphate group. In the current study a negative effect on SBS was found in the group with the acid surface treatment as mentioned previously. In this context, the mechanisms of acid environment in the MDP-zirconia reaction needs to be further researched, since the affinity to form hydrogen bonding of some acids with ZrO₂ powder has been previously reported²⁵). A study proposed that an alkaline surface treatment can improve the SBS, in addition, found statistical significance while comparing neutral acid and alkaline pH groups, in which the alkaline surface treatment group (pH 11–12) had the highest SBS even after 30 days of water storage¹⁶). In our study the higher MPa value was computed for alkaline surface treatment group, but there was no significant difference in the SBS value when compared with APA group, and it is consistent with the results of a different study, where consisted in surface chemical treatment with primers containing MDP-10 monomer and 0.5 M NaOH in single or combined treatments, and they found an improved SBS for groups that combined NaOH and MDP-10-containing primer. However, no significant differences were found in that study¹⁷). Differences in the results of alkaline surface treatment between these researches can be attributed to the difference in the pH or M concentration in each of the studies, hence supporting the idea that SBS improvement could be pH value-dependent. However other research compared four different alkaline coatings with distinct pH values, and they concluded that higher alkaline pH values used before MDP conditioning would not necessarily improve bonding²⁶). Further studies should be made to determine a specific pH range to concisely improve the SBS as alkaline surface treatment could be a promising alternative to improve adhesion systems even in biological applications.

For ARI evaluation, we found that 63.3% of the fracture mode in APA and alkaline groups, belong to score 1, which means that there is a cohesive failure within the resin cement material in some segment, suggesting that SBS between resin cement and zirconia is higher than the material itself. We also found a score of 0 for acid group that is equal to adhesive failure mode in all the samples, which means that there is no mechanical interlocking or chemical adhesion between zirconia and the luting agent.

In accordance with the results of this study, we can suggest that roughness and MDP monomers are not the only success key in zirconia adhesion, chemical surface treatment different to adhesive monomers also plays an important role in the process; we found that alkaline solution improves SBS values, while acid solutions have a negative effect. Meanwhile, acid solutions for surface treatments had been suggested for zirconia surface modification prior adhesion, it must be taken in account that zirconia luting agents are usually dual

cure materials due to zirconia opacity and they have an initial chemical polymerizing process which could be affected by pH.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions can be drawn:

1. The surface treatment increased the roughness of zirconia and changed its morphology due to treatment with APA, HF acid, and sodium hydroxide.
2. The differences between treated surfaces do not have significant effects on surface roughness.
3. SBS was significantly affected by surface treatments with acid solution, whereas alkaline treatment with NaOH slightly improved SBS values.
4. The adhesive failure mode for HF group confirmed the negative effect of acid treatment on zirconia adhesion with dual cured resin cement.

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CONFLICT OF INTEREST

Authors declare that they have no conflict of interest.

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