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ORIGINAL RESEARCH

Evaluation Of The Effect Of Different Surface Treatments, Aging And Enzymatic Degradation On Zirconia-Resin Micro-Shear Bond Strength

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Purpose: The purpose of this study was to evaluate the effect of surface treatments on zirconia-resin bonding and the effect of aging on bond durability for one year.
Method: Three hundred and twenty zirconia blocks were divided into 4 equal study groups. Group 1 (control): as-sintered, group 2: (GB): grit-blasted, group 3: (LAS): laser-etched, group 4: (SIE): selective infiltration etching. Composite cylinders were bonded to the zirconia with resin cement and ceramic primer. Aging was performed following 3 different aging protocols: thermocycling, storage in distilled water, or storage in an enzymatic esterase solution. Micro-shear bond strength test (μSBS) was recorded using a universal testing machine. μSBS values were analyzed using two-way Analysis of Variance followed by Tukey post-hoc tests. Level of significance was set at 0.05.
Results: GB, LAS and SIE groups showed significantly higher values when compared to control. Groups GB, LAS and SIE reported a significant decrease up to 50% in μSBS after water storage and enzymatic degradation, while control group reported a 90% decrease. Failure analysis showed mainly adhesive failure for control group, while the percentage of cohesive failure in resin cement was higher in SIE group compared to GB and LAS groups.
Conclusion: Water aging and esterase solutions played a significant role by increasing bond degradation. A minimum of one-year water and esterase storage medium should be used to evaluate the durability of the bond between resin cement and zirconia.
Keywords: air abrasion, laser, MDP primer, resin cement, aging

Introduction

The advancement of digital dentistry has expanded applications of zirconia in prosthetic dentistry.[1] The superior mechanical and esthetic properties combined with biocompatibility[2] make zirconia the best substitute for ceramic fused to metal restorations.[3] However, the adhesive bond to zirconia is questionable in some clinical situations, such as short abutment, compromised retention, and resin-bonded fixed partial dentures.[4] This is related to the silica-free composition that characterizes zirconia as an acid-resistant material and renders hydrofluoric acid etching used on silica-based ceramics ineffective on zirconia.[5,6]

Different roughening techniques are suggested to promote adequate retention between resin cements and zirconia. The most common is grit-blasting with aluminum oxide (Al_2O_3) particles using different particle shapes and sizes, and different abrasive times and pressures.[7,8] Grit-blasting produces a micro-roughened zirconia surface that enhances the adhesive bonding by increasing surface energy, wettability and surface area.[9] However, the stress exerted by grit-blasting may lead to cracks on the zirconia surface and further induce tetragonal to monoclinic phase transformation. The volume expansion of the transformed

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grains induces compressive stresses at the crack tip thus preventing crack propagation with an increase in mechanical strength. This phenomenon is known to as transformation toughening[10] but on the other hand, excessive amounts of transformed grains tend to decrease mechanical strength[11] and may affect the long-term performance of zirconia restorations.[12,13]

Alternative techniques for zirconia surface conditioning have been introduced such as grit-blasting before sintering,[8] selective infiltration-etching technique, tribochemical silica coating,[15] plasma spraying,[16] surface fluorination,[17] laser treatments,[18] and silica coating.[19] Silica coating provides a siliconized zirconia surface that can chemically interact with the applied silane to increase the adhesive bond to resin cements.[20] The durability of adhesive bonding to zirconia restorations relies on the mechanical and chemical surface treatment modalities.[21] Studies have shown that chemical bonding between resin cements and zirconia surfaces could be achieved by using a primer and a resin cement based on adhesive monomer containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) acting as a coupling agent.[22,23] However, despite the fact that grit-blasting could affect the final performance of zirconia restoration when subjected to oral stresses,[2,24,25] the use of laser as an alternative surface treatment method is now of interest and is reported in numerous studies.[26,27] Nd-YAG laser surface treatments have shown increased surface roughness, wettability and bond strength to resin cement,[28] but due to the different laser types and different parameters used, these studies have shown variable results regarding bond strength.[29]

Another important factor affecting the clinical performance of a restoration is aging. Oral mechanical stresses, temperature, and humidity have a great influence on bond degradation of zirconia restorations.[21,25] In several studies, thermo-cycling and varying times of water-storage have been used as an artificial ageing method.[30–33]

The aims of this study were to evaluate 1)- different zirconia surface treatments and their effect on micro-shear bond strength of resin to zirconia and 2)- the effect of aging on the resin–zirconia interface. The null hypotheses tested were that there was no significant difference between the different surface treatments evaluated, and there was no significant decrease of the initial micro-shear bond strength values after different aging methods.

Materials And Methods

Sample Preparations

A total of 320 pre-sintered quadrangles were cut out of zirconia blocks (Amman Girrbach, Koblach, Austria) with the dimensions of 10×10×3 using a low-speed diamond saw (Buehler, Lake Buff, WI, USA) under running tap water. Specimens were polished with silicon carbide grit paper #400 (Grit flex, Italy) using a polishing machine (Buehler, Lake Buff, WI, USA) for surface standardization,[34] and then subjected to oil-free high-pressure airflow for 1min. Samples were then sintered in a sintering furnace (Ceramil Therm, Amman Girrbach, Austria) according to manufacturer's instructions.

Surface Treatment Protocols

Samples were randomly divided into 4 study groups (n=80) according to the surface treatment performed. Group 1 (control): as-sintered; group 2 (GB): the surfaces of sintered samples were grit-blasted with 50µm alumina particles (Vacumat 300, Vita Zahnfabrik, Germany) for 15s under 3.5 bars pressure at a working distance of 10mm; group 3 (LAS): surfaces were etched using Er,Cr:YSGG laser (Waterlase MD system, Biolase, San Clemente, CA, USA) for 2mins (5.5W power, 20Hz with 100mJ energy).[35] A custom-made computerized robot was used to standardize the application of laser (Figure 1);[36] group 4 (SIE): surfaces were subjected to selective infiltration etching (SIE) technique.[14] After sintering, the surface of the specimens was abraded by a sequence of grit papers ≠ 200–800. A thin layer of low-fusing melting glass was applied on the zirconia surface. The specimens were then heated to 750°C for 2mins, cooled to 650°C for 1min, reheated to 750°C for an additional 1min, and then cooled to room temperature. The specimens were immersed in a bath of 9% hydrofluoric acid for 20mins, and then every specimen was subjected to a high stream of air and water for 2mins. All samples were cleaned using an ultrasonic device for 10mins and left to dry at room temperature for 24hrs before bonding.

Composite Cylinders Preparation And Cementation

A total of 320 resin composite cylinders (2mm diameter and 2mm height) were prepared by packing the resin composite (Z250, 3M ESPE, Saint Paul, MN, USA) into a Plexiglas mold. 4mm Plexiglas slabs was placed on the surface and the bottom of composite to ensure a flat surface and to prevent the oxygen

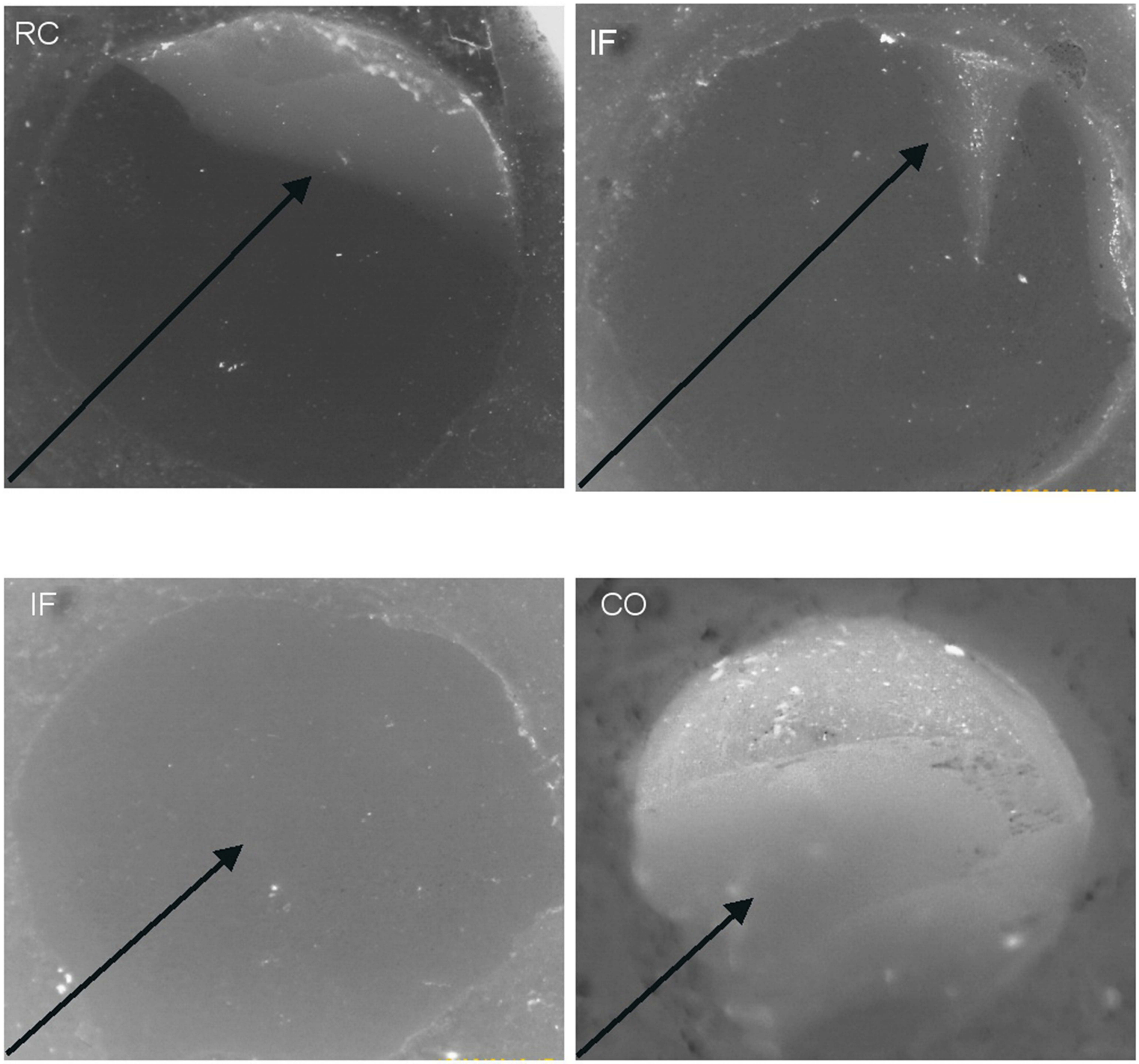

Figure 1 Failure modes of the studied groups: Cohesive failure (**RC**) within the resin cement; a part of resin cement adhered to zirconia surface (black arrow). Interfacial failure (**IF**) between the cement and zirconia; a little or no resin cement adhered on the zirconia surface (black arrow). Cohesive within the resin composite (**CO**); the fracture with the composite cylinder (back arrow).

inhibition layer. Light-curing was done for 40s from the top and 40s from the bottom (Elipar Free Light 2 LED, 3M-Espe, wave length 430–480nm, light intensity 1000Mw/cm^2, Saint Paul, Minnesota, USA). A primer (Clearfil Ceramic Primer, Kuraray, Tokyo, Japan) containing MDP was applied on the zirconia surfaces and left to dry for the 20s. The composite micro-discs were bonded to the treated zirconia surface using a dual-cure resin cement (Panavia F2.0, Kuraray Dental, Tokyo) under a fixed load of 450g. Excess cement was removed gently using a curette and micro-brush, and cement was light-cured from 2 lateral sides and the top for 40s each using the same light-curing device.

Aging

The 80 specimens in each group were then divided into 8 sub-groups (n=10) according to the aging treatments: Sub-group 1 (control): no any aging performed, sub-group 2 was tested after thermocycling (5°C-55°C,

Table 1 Mean In MPa And Standard Deviation (values) Of *μSBS* Of Tested Groups Subjected To Different Surface Treatments And Aging

Groups	Control	TCL	Water			Esterase		
	No storage		2 Weeks	24 Weeks	48 Weeks	2 weeks	24 Weeks	48 Weeks
Control	10.4 (7.2)$^{a, A}$	6.2 (3.1)$^{b, A}$	6.8 (3.7)$^{b, A}$	2.6 (2.9)$^{c, A}$	0.2 (0.7)$^{d, A}$	9.4 (3.6)$^{a, A}$	4.3 (1.6)$^{b, A}$	1.2 (0.4)$^{c, d, A}$
GB	28.3 (3.2)$^{a, B}$	24.4 (3.4)$^{a, B}$	25.2 (5.4)$^{a, B}$	20.7 (5.9)$^{a, b, B, C}$	17.3 (4.2)$^{b, B}$	21.1 (7.6)$^{b, c, B}$	17.5 (4.4)$^{b, c, B}$	13.2 (3.9)$^{b, d, B}$
LAS	25.9 (3.8)$^{a, B}$	22.3 (3.6)$^{a, B}$	20.5 (4.3)$^{a, B}$	14.8 (4.7)$^{b, B}$	14.8 (4.7)$^{b, B}$	21.1 (3.7)$^{a, B}$	15.0 (4.2)$^{b, B}$	11.8 (3.4)$^{c, B}$
SIE	36 (4.3)$^{a, C}$	32 (4.2)$^{a, c}$	30.2 (8.9)$^{a, C}$	28.2 (8.1)$^{a, C}$	22.9 (8.3)$^{b, B}$	34.6 (4.1)$^{a, C}$	30.7 (4.7)$^{a, C}$	24.2 (8.2)$^{b, c}$

Notes: Similar small superscripts indicate no significant difference between storage media for the same surface treatment (horizontal comparison). Similar capital superscripts indicate no statistical significance between the surface treatments (vertical comparison).

20sec dwelling time, 10,000 cycles), sub-groups 3, 4 and 5 were tested after storage in distilled water for 2, 24 and 48 weeks, respectively, sub-groups 6, 7 and 8 were tested after storage in a prepared 0.1unit/mL enzymatic esterase solution[37] for 2, 24 and 48 weeks, respectively (Table1).

Microshear Bond Strength Test

The microshear bond strength test (μSBS) was performed using a custom-made uni-bevel semi-circle chisel-shaped indenter mounted on a universal testing machine (YL-UTM Main, YLE GmBH, Bad Koenig, Germany) at a crosshead speed of 0.5mm/min until failure. The resin to zirconia shear bond strength values was obtained by dividing the load of failure by the bonded area.

Failure Mode Analysis

After μSBS testing, zirconia surfaces were examined under optical microscope at ×20 magnification (BH-2, Olympus, Tokyo, Japan). Failure modes were classified as an interfacial failure (IF) where the crack traveled at the zirconia-resin cement interface with consideration of the area of crack origin, a cohesive failure (RC) in the resin cement where the crack originated outside the bonded interface and cohesive within the resin composite (CO) (Figure 1).[37] The percentage of failures based on failure modes (IF, RC, and CO) are shown in Table 2.

Statistical Analysis

The data were analyzed using a statistical software package (SPSS version 23, Armonk, NY, USA). After normality and homosexuality confirmation, two-way Analysis of Variance was conducted followed by Tukey post hoc tests for multiple comparisons. The level of significance was set at 0.05.

Results

The data reported in Table 1 show a significant effect of surface treatments ($p<0.05$, F=142.3), as well as aging and enzymatic degradation ($p<0.05$, F=43.8) on zirconia-resin bond strength ($p<0.05$, F=5.6). GB, LAS, and SIE groups displayed significantly higher bond strength compared to

Table 2 Failure Type And Percentage Of Fracture Of Zirconia And Resin Composite Discs

Groups	Control	TCL	Water			Esterase		
	No Storage		2 Weeks	24 Weeks	48 Weeks	2 Weeks	24 Weeks	48 Weeks
CON	IF 100%	IF 100%	IF 100%	IF 100%	IF 100%	IF 100%	IF 100%	IF 100%
GB	IF 60% RC 40%	IF 70% RC 30%	IF 80% RC 20%	IF 90% RC 10%	IF 100%	IF 90% RC 10%	IF 100%	IF 100%
LAS	IF 70% RC 30%	IF 80% RC 20%	IF 80% RC 20%	IF 100%	IF 100%	IF 80% RC 20%	IF 100%	IF 100%
SIE	IF 20% RC 30% CO 50%	IF 30% RC 30% CO 40%	IF 40% RC 40% CO 20%	IF 60% RC 40%	IF 70% RC 30%	IF 50% RC 40% CO 10%	IF 80% RC 20%	IF 90% RC 10%

Abbreviations: IF, interfacial; RC, cohesive in resin cement; CO, cohesive in resin composite.

the control group. Groups that received surface treatment reported a significant reduction up to 50% in bond strength when subjected to thermocycling, water storage and enzymatic degradation as compared to 90% decrease of bond strength that was reported in the no surface treatment group. GB and LAS groups displayed a significant difference at 24 and 48 weeks of water storage, and significant differences were reported between the SIE group and all other groups at different storage times (Table 1). A significant decrease in bond strength was noted in GB and LAS groups at 48 weeks of water and esterase storage while SIE group was the least affected by aging.

Failure mode analysis showed complete interfacial failure for the control group, while the percentage of cohesive failure in resin cement was higher in SIE group compared to GB and LAS groups. At 48 weeks, GB and LAS groups reported a complete interfacial failure while SIE group showed the highest interfacial percentage failure at 48 weeks in esterase storage (Table 2).

Discussion

Different zirconia surface treatments were investigated in the present study to optimize the surface for micromechanical and chemical interaction with the adhesive system. Since the initial μSBS values of experimental groups were significantly higher than the control, the first null hypothesis had to be rejected. The surface treatment of the samples was required to improve the mechanical interaction with the resin cement.[38] Ozcan et al,[39] stated that MDP monomer bonded directly to metal oxides, and that the hydroxyl group in MDP monomer reacted with the hydroxyl group on the zirconia surface. Nevertheless, Yi et al[40] reported that the amount and flow of functional monomer alone were not sufficient to increase the zirconia adhesion ability without any surface pretreatment. In the same manner, several authors[41–43] confirmed that the durable bond to zirconia ceramic could not be achieved with MDP containing cement without surface treatment.

The thermal stresses and humid environment inside the oral cavity may exhibit subcritical crack growth and hydrolysis of the resin at the bonded interface.[44] Many studies[45–47] used a combination of thermal cycling and water storage to test the resin bond durability; nevertheless, the best aging method remains a controversial topic. Hallmann et al,[45] stated that water storage with additional thermocycling did not decrease bond strength value, whereas Heikkinen[46] and Qeblawi et al[47] concluded that the combination of water storage and thermocycling decreased bond strength values significantly. In this study, thermocycling and water storage were investigated in different combinations in an attempt to evaluate the most effective aging method.[31,48,49] The μSBS values obtained were significantly different after each aging method. The use of 10,000 thermocycles showed no significant decrease of μSBS values in any group except for the control group. The failure mode of the control group showed a 100% interfacial failure, whereas the mode failure percentage of other groups showed no difference. Water storage, however, appeared to be more influential when it comes to evaluate bond degradation since the results showed that μSBS values of all groups declined significantly after 6 months with another significant decrease after 12 months of water storage. As well as, the interfacial failure became 90% after 6 months and 100% after 12 months for GB and LAS groups and 70% for SIE group.

The present results are in accordance with Aboushelib et al,[50] who reported that the initial bond strength was not stable after water storage for 3 months, and with Oyagüe et al[51] who concluded that water storage for 6 months played an important role in zirconia resin bond deterioration. Since human saliva has a greater ability than water to degrade resin.[52] Esterase solution was used in this study as a storage medium to improve extrapolation of the results to the clinics, despite the fact that it was used as a contamination medium in other studies[53–55] Our results showed a significant decrease of μSBS values at 6 months of esterase storage and another significant drop after 12 months. The only surface treatment that preserved a high bond value was SIE. The interfacial failure of GB and LAS groups was 100% after 6 and 12 months, and 90% for SIE group after 12 months. The present results accord with a recent study by Aboushelib et al,[56] that concluded that groups treated by alumina and laser had their μSBS values reduced by 50% after 12 months of esterase storage whereas the best results were for the groups stored in artificial saliva with minor decrease in bond over the same period. This may reveal that the achieved bond for GB and LAS groups did not resist the water and esterase hydrolysis, even with the use of phosphate monomer as an adhesive promoter.

Many studies stated that the MDP monomer reduced the hydrolysis of bond,[21,45,57,58] as it prevents the hydrolytic effect of the cement layer through the bonded margin.[59] However, other studies that used MDP monomer stated that the bond did degrade after a period of water storage and thermocycling.[46,47,51]

Based on the results of the present study, we can conclude that there is no one factor responsible for bond degradation. The μSBS values obtained during an experiment are a direct function of the combination of: 1- A suitable surface treatment method and the use of and MDP-based primer (that will dictate the quality of the resin-zirconia bond), and 2- The type of aging treatment used is paramount for the validity of in-vitro testing. It is noteworthy that thermocycling at 10,000 cycles did not degrade the bond. Further research using higher cycles values should be done to prove or disprove the effect of thermocycling on μSBS values. 3-A minimum of one year in storage medium should be used during aging tests. Further studies should be performed to confirm that fact.

Conclusions

Within the limitation of this study the following conclusions may be drawn:

1. The primer used can lead to a sufficient adhesion between zirconia and resin cement.
2. A minimum of one-year water and esterase storage medium should be used to evaluate the durability of bond between resin cement and zirconia.
3. Water aging and esterase solutions played a significant role by increasing bond degradation

Disclosure

The authors report no conflicts of interest in this work.

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