Original Article

Evaluation of Shear Bond Strength of Zirconia Based Materials on Veneering Ceramics after Surface Treatment with Liner and Different Etching Agents” an *In Vitro* Study

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Abstract

Background Data: Zirconia framework, the most commonly used esthetic restorative biomaterial has the debonding that leads to either chipping or delamination of the veneering ceramic. This study uses a combination of surface treatment including liner and etching agents to increase the bond strength. **Objective:** The aim of the study was to evaluate the effect of different surface treatments using sandblasting, liner, etching agents, and combination of surface treatment on shear bond strength (SBS) of zirconia to veneering ceramic. **Materials and Methods:** One hundred and fifty zirconia discs of the 10 mm diameter and 18 mm height were prepared using computer-aided design and computer-aided machine, Specimens were divided into five groups ($n = 30$) according to surface treatment method; group 1: control, group 2: liner application (Emax ceam zirliner), group 3: Sandblasting + liner application, group 4: Hydrofloric acid (HF) + liner application, group5: potassium hydrogen difloride (KHF2) + liner application, SBS was tested using universal testing machine with a crosshead speed 1 mm/min. SBS was calculated consistent with equation: SBS (MPa) = load (N)/area (mm^2). The data were analyzed using analysis of variance test and Bonferonis test. **Results:** Mean SBS is highest for the group KHF2 + liner (43.31 ± 9.25) followed by the group HF + liner (39.01 ± 11.14), sandblasting + liner (33.75 ± 8.76), Control (32.9 ± 8.92) and liner (30.46 ± 8.80). **Conclusion:** This study shows that KHF2 + Lliner, HF + liner application on zirconia substructure increased bond strength to veneering ceramic compared with other groups.

Keywords: Air particle abrasion, Liner, Hydrofloric acid, Potassium hydrogen difloride, Shear bond strength, Zirconia.

Introduction

Zirconium dioxide (zirconia) has become more widely used for ceramic restorations and in clinical practice because of its outstanding mechanical properties, chemical stability, biocompatibility, and aesthetics. Pure zirconia has a monoclinic phase at room temperature, but the addition of 3 mol% yttria creates yttria-stabilized tetragonal zirconia polycrystal (Y-TZP).[1]

Retention of the tetragonal phase at room temperature provides better mechanical properties. Thus, Y-TZP has drawn attention recently as a core material for all-ceramic restorations. Although zirconia Y-TZP materials are more translucent than metal alloys it has drawback of being essentially white and opaque. Thus, zirconia-based restorations are usually covered by a veneering ceramic for enhanced esthetic quality.[2,3]

The mechanical integrity and adhesion are key factors in the success of veneer/core bilayered restorations. Delamination (failure at the porcelain/zirconia interface) and chipping (failure within the veneering porcelain) are the two most common failure modes.[4,5]

Failures in zirconia-based restorations either originate from the veneer and propagate to the interface or originate from the zirconia/veneer interface. Voids and flaws inevitably exist at the interface, and crack may initiate from these voids and flaws due to stress concentration under a certain loading.

To resolve this problem, researchers have explored various surface treatment methods to improve the bond strength

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between zirconia and ceramic layering, such as airborne particle abrasion, liner application, laser treatment, elective infiltration etching, and silica coating, acid etching.[6,7]

Liners are proprietary ceramic materials that are suggested by some manufacturers to be applied as an intermediate layer between the zirconia core and the veneer to maximize bond strength, shade effects, fluorescence, and to increase the wetting property on the zirconia surface.[8]

Hydrofluoric acid (HF) treatment is commonly used on silica-based ceramics to react and remove the glassy matrix that contains silica. It creates surface roughness, results in enhanced wettability and surface energy on the ceramic surface, which allows greater penetration of the resin cement tags, increasing the bond strength between the ceramic and cement.[9,10] and recently some studies have shown that etching with potassium hydrogen difluoride (KHF2) was effective for zirconia. It will cause roughness and wettability for zirconia and it may influence on the bonding ability.[11]

However, extensive investigations of approaches and combinations of surface treatments are lacking still. Thus the objective of the present study was to evaluate and compare the effects of the combination of liner and acid etching surface treatments on shear bond strength (SBS) of zirconia to veneering ceramic.

Materials And Methods

A stainless-steel mold with the dimensions of 10 mm diameter and 18 mm height was prepared and mounted in a holder. The mold was scanned with computer-aided design and computer-aided machine (CAD/CAM) scanner (Amanngirrbach Germany). And this model was used to fabricate 150 discs of the same size from pre-sintered zirconia blocks. Zirconium oxide blocks were milled according to the manufacturer's instruction. Then, they were cleaned, dried, and sintered according to the suggested firing schedules.

The bonding surfaces of zirconia core specimens were polished with diamond paste using an ascending stepwise approach starting with 120 grit and ending with 800 grit silicon carbide paper, 120, 240, 320, 400, 600, and finally 800, using a rotating metallographic polishing device under a fixed load and water cooling. This was done to obtain standardized surface roughness. All the specimens are thereafter sintered at 1500°C for 8 h according to manufacturer instructions.

Surface treatment methods

150 zirconia discs were randomly divided into five groups according to the surface treatment methods:

Group1: Control (Sandblasting 50/30 microns and 3.5 bar pressure)

Al_2O_3 airborne-particle abrasion was performed using a sandblast machine with 50/30 μm particle size for 15 s at a 10 mm distance from the surface and with a pressure of 3.5 bar. Finally, the samples were ultrasonically cleaned in 96% isopropyl alcohol for 3 min and steam-cleaned for 15 s.

Group 2: Liner

Ceramic liner (e max ceram zirliner) application according to manufacturer instructions.

Group 3: Sandblasting + liner

Al_2O_3 airborne-particle abrasion was performed using a sandblast machine with 15 μm particle size for 15 s at a 10 mm distance from the surface and with a pressure of 1.5 bar. Finally, the specimens were ultrasonically cleaned in 96% isopropyl alcohol for 3 min and steam-cleaned for 15 s followed by ceramic liner application according to manufacturer instructions.

Group 4: Etching (HF) + liner

Zirconia specimens were immersed in 9.5% HF at 25 C for 1 h. Subsequently, ceramic liner application was done according to manufacturer instructions.

Group 5: Etching (KHF2) +liner

All zirconia specimens were powder coated with KHF2 in the amount of 70 mg. The specimens were then heated in a porcelain furnace to a temperature of 280°C. The bonding surface was then cleaned for 15 s using a steam cleaner for 15 s followed by compressed air for 15 s. Finally, ceramic liner application was done according to manufacturer instructions.

Application of porcelain

Using a specially designed, separable stainless-steel mold, a prepared zirconia disk specimen was placed in the mold where clearance of 10 mm diameter and 3-mm height was available above the core material for condensing the veneer ceramic. The veneering procedure was performed using the manual layering technique. First, two-liner layers of porcelain were applied and fired independently, then the dentin porcelain was condensed using the vibration blotting technique, fired and finally, a glaze firing was performed according to the manufacturer's instruction.

All specimens were stored in distilled water at 37°C for 1 week. And then thermal cycled for 5000 cycles, 5–55°C with a 30 s duration.

SBS test

Then, these metal holders were mounted in universal testing machine. Specimens were tightened and stabilized to ensure that the 1-mm thick edge of the shearing device is in contact with the core surface and was positioned as close as possible to the veneer-core interface. Shear load was applied at a crosshead speed of 0.5 mm/min until fracture occurred. The ultimate load to failure was recorded in Newton (N). The average SBS (MPa) was calculated by dividing the load (N) at which failure occurred by the bonding area (mm^2) as follows:

Shear stress (MPa) = Load (N)/surface area

Statistical analysis

The SBS values between the zirconia core and the veneer ceramic were evaluated with one-way analysis of variance (ANOVA). Post-hoc Bonferroni test was applied for inter-group comparison. Results are displayed in the form of mean values with standard deviations for each parameter combination and were considered statistically significant when $P < 0.05$.

Results

One-way ANOVA revealed significant difference between the groups regarding the mean bond strength, i.e., $P = 0.001$ (Table 1). The post hoc Bonferroni analysis is done to find out the significant difference between any of two given groups. Results showed stastically significant difference between the groups control and KHF2 + liner ($P = 0.00$), liner and HF + liner ($P = 0.006$); liner and KHF2 + liner ($P = 0.00$); sandblasting + liner and KHF2 + liner ($P = 0.001$) (Table 2). Mean SBS is highest for the group KHF2 + Liner (43.31 ± 9.25) followed by the group HF + liner (39.01 ± 11.14), sandblasting + liner (33.75 ± 8.76), Control (32.9 ± 8.92) and liner (30.46 ± 8.80) (Graph 1).

Discussion

Bond strength of zirconia with veneering ceramics is influenced by many factors like strength of chemical bonds, mechanical interlocking, type and concentration of defects at the interface, inappropriate framework support for the layering porcelain, wetting properties, and the degree of residual compressive stress in the veneering layer due to a difference in the coefficient of thermal expansion (CTE) between zirconia and the veneering ceramic.[2,3]

Clinical studies show that the main mode of failure of zirconia prosthesis was the chipping or delamination of veneering ceramic from the zirconia core. Thus, in recent past, focus has been to extend the bond strength between zirconia and veneering ceramic. Several *in vitro* studies are performed using different surface treatments such as sandblasting, liner application, silica coating, laser, and acid etching.[4,5] In an effort to get more efficient surface treatment, contemporary methods like liner application and acid etching treatment were adopted and have shown promising results.[10,11]

Thus, the present study uses a combination of surface treatments using liner application and acid etching to evaluate its effect on the SBS of zirconia to veneering ceramic with sandblasting as positive control group. In this study, the results have shown statistically significant difference between groups with highest bond strength values for the group treated with KHF2 + liner (group 5) and HF + liner (group 4) application followed by sandblasting group. Thus, the null hypothesis is rejected.

Aboushelib *et al*., in their study, demonstrated that type of zirconia framework used could affect the bond strength, and they found that increased bond strength obtained in CAD/CAM milled zirconia. This was due to the formation of trace lines by CAD/CAM milling and consequent erasing by air abrasion leading to increased roughness and ultimately increasing the bond strength between zirconia and veneering ceramic. Thus, the present study utilizes CAD/CAM milled zirconia blocks for surface treatment.[12,13]

Table 1: Comparison of the mean shear bond strength among the groups using ANOVA

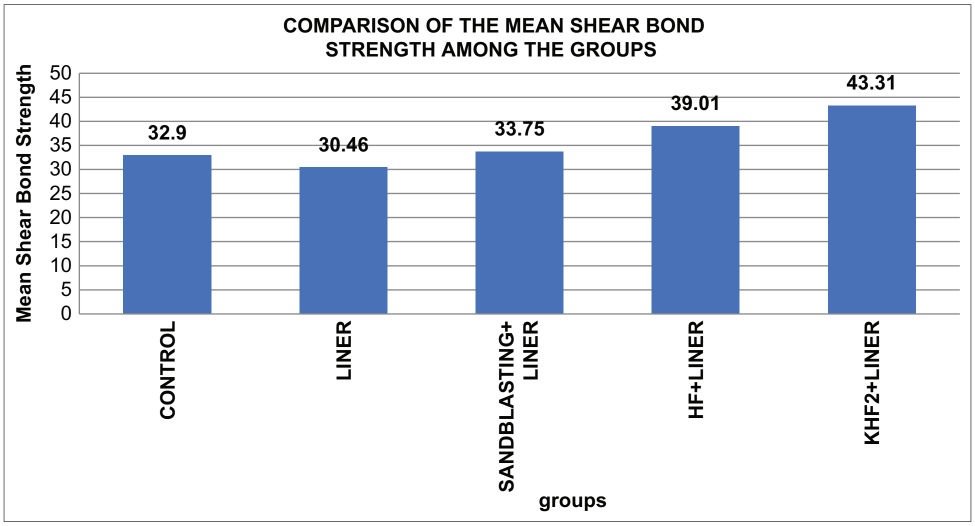
Groups	*n*	Minimum	Maximum	Mean	S.D	F-value	*P*-value
Control	30	15.4	48.5	32.90	8.92	9.10	0.00*
Liner	30	18.4	48.2	30.46	8.80		
Sandblasting+liner	30	17.6	47.5	33.75	8.76		
HF+Liner	30	24.0	54.9	39.01	11.14		
KHF2+Liner	30	29.5	60.5	43.31	9.25		

HF: Hydrofloric acid, KHF: Hydrogen difloride

Table 2: Inter-group comparison of shear bond strength using *post-hoc* bonferroni

Group	Groups	Mean Difference	*P*-value	95% Confidence Interval	
				Lower Bound	Upper Bound
Control	Liner	2.44	1.000	–4.49	9.37
	Sandblasting+Liner	–0.850	1.000	–7.78	6.08
	HF+Liner	–6.11	0.130	–13.04	0.82
	KHF2+Liner	–10.40	0.000*	–17.34	–3.47
Liner	Sandblasting+Liner	–3.29	1.000	–10.22	3.64
	HF+Liner	–8.55	0.006*	–15.48	–1.62
	KHF2+Liner	–12.84	0.000*	–19.78	–5.91
Sandblasting+Liner	HF+Liner	–5.26	0.321	–12.19	1.67
	KHF2+Liner	–9.55	0.001*	–16.49	–2.62
HF+liner	KHF2+Liner	–4.29	0.796	–11.22	2.64

HF: Hydrofloric acid, KHF: Hydrogen difloride



Graph 1: Mean Shear Bond Strength

Sandblasting is said to be the foremost effective surface treatment in increasing the bond strength by roughening the surface of zirconia ultimately causing micromechanical interlocking between zirconia and the veneering ceramic. Thus, sandblasting has been included as positive control for the study.

Few studies have reported liner and acid etching to be effective for increasing the bond strength. Liners are ceramic materials applied as an intermediate layer between the zirconia core and the veneer to maximize bond strength, shade effects, fluorescence and wetting property on the zirconia surface.[13,14]

Strong discrepancy in CTE between veneering ceramic and zirconia affect their bond strength. When there is phase transition from monoclinic to tetragonal phase, 0.6% of zirconia is said to retain its monoclinic crystal phase. These residual crystals in the monoclinic phase have a CTE close to that of liner. Thus, the application of a thin layer of the liner with slow cooling process causes a decrease in residual stresses in ceramic material. Thus, liner was included in the present study.

However, in the present study, liner application gave a bond strength value lesser than the other groups where the combination of surface treatment was carried out. This is agreement with the study Fischer *et al*. that the use of a liner alone as less significant effect on bond strength.[15] Ruyter *et al*. reported that etching with KHF2 chemically changes the zirconia to fluoride compounds K2(ZrF6), resulting in sediments containing ZrF4 grains. This acts as porosities and surface irregularities providing better surface for interlocking with luting materials. which would improve bond strength compared to sandblasting.[16]

Undercuts created by etching procedure using KHF2 were more effiecient than those created by alumina blasting. This is because undercuts created by etching are deeper and more retentive for bonding. The present study proves the highest bond strength with combined use of liner with KHF2. This might be due to combined advantage of using liner which reduces the residual stress and KHF2 which causes retentive areas for better bonding.

Dental porcelain consists of glassy and crystal phases, HF dissolves glass by reacting with silicon oxide and leaves the crystalline phase, creating surface roughness and increases the bond strength. The present study shows second-highest bond strength for the group where HF acid etching was done followed by liner application. The mechanism could be etching will left microporosities so thereby it will aids in the retention and better bonding.

Thus, this study is to evaluate the bond strength of zirconia with various surface treatments with combination. This discrepancy in results may be attributed to differences in combination of surface treatment methods. It utilizes the advantages of liner application and acid etching roughness properties.

Conclusion

Within the limitations of this *in vitro* study liner application alone is less effective, The KHF2+ liner application and HF + liner application showed increase bond strength between zirconia and veneering ceramic. Considering the multifactorial and complex nature of the fracture behavior of veneered zirconia restorations, *in vivo* studies are still required to better understand the clinical significance of the findings of this *in vitro* study.

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