ORIGINAL RESEARCH

Effect of Various Surface Treatments of Zirconia on its Adhesive Properties to Dentin: An *In Vitro* Study

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Abstract

Aim: To assess the effectiveness of various surface treatments and adhesives on the bond strength of zirconia-based ceramic to dentin.

Materials and methods: Eighty samples of zirconia were subjected to the four-surface treatment protocols (sandblasting, 48% hydrofluoric acid (HF), 48% hydrofluoric acid + 70% nitric acid (HNO_3) and no treatment (control) following which the samples from each group were subdivided into two subgroups ($n = 10$) based on the resin cement employed for cementation (RelyX U200 and G-Cem Linkforce). The bonded specimens were subjected to shear stress to measure the bond strength using Universal testing machine. To test the difference in bond strength among the eight study groups, the Kruskal–Wallis ANOVA test was applied and for comparison between cements in each group, Mann–Whitney *U* test was applied.

Results: The highest bond strength values were observed for 48% HF group cemented with G-Cem Linkforce resin cement (16.220 ± 1.574) and lowest for control group–RelyX (4.954 ± 0.972). G-Cem cement showed higher bond strength than RelyX for all surface treatments except 48% HF + 70% nitric acid.

Conclusion: It can be inferred that 48% HF can etch zirconia and generate a porous structure that proves to be beneficial for bonding.

Clinical significance: The increasing demand for esthetics has led to the replacement of metal-ceramic materials with zirconia-based ceramics. However, the chemical inertness of zirconia to various conventional surface treating agents has continuously challenged researchers to discover a new surface treatment protocol that could enhance the bond strength of zirconia.

Keywords: Bond strength, Resin cements, Sandblasting, Zirconia.

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Introduction

The increasing requirement for esthetics has guided dental professionals to prefer all-ceramic materials over metal and porcelain-fused metal-ceramic restorations for oral rehabilitation which can be attributed to its advantageous optical properties like translucency, biocompatibility, low thermal conductivity as well as low wear resistance, and low abrasiveness.[1] However, the applications of all-ceramic restorations are limited due to their brittleness, sensitivity to flaws and defects, low tensile strength, and fracture toughness.[2] Recently, high-strength zirconia ceramics have become a highly recommended material in dentistry because of their favorable characteristics such as high flexural strength and toughness, esthetics, and biocompatibility.[3] But adhesion to the tooth structure poses a challenge to every clinician as it is a polycrystalline material that is resistant to conventional etching techniques due to its silica-free structure.[4] Many chemical and mechanical pretreatment methods for zirconia surfaces have been researched including sandblasting, hydrofluoric acid (HF) etching, the use of a combination of hydrofluoric acid and nitric acid (HNO_3) tribochemical silica coating. However, an optimal resin bonding technique for zirconia has not been established yet. Air abrasion with alumina particles often known as sandblasting, roughens the zirconia surface and aids in resin/ceramic micromechanical interlock formation thereby increasing the bond strength. However, since the microporosities created by sandblasting may act as crack-initiators thereby weakening the ceramic restoration, it is imperative to explore new methodologies that may enhance the long-term results and do not interfere with the properties of ceramics.[5] Several studies have reported conflicting results on the increase and decrease in bond strength after sandblasting.

Acid etching enables the homogenous roughening of material regardless of its size and shape. Recently, various studies investigating the effectiveness of HF were found to be beneficial for treating the zirconia surface and thus improving the resin-to-zirconia adhesion.[5–7] As demonstrated by Yen et al.,[6] HF reacts preferentially with the silica phase in a glassy matrix to form hexafluorosilicates rendering the surface of the ceramic rough. Concentrations ranging between 4 and 10% HF acid are commonly used in a dental laboratory. However, various studies have proved that higher concentrations of etchant can modify the surface morphology of zirconia by creating a highly uneven surface.[7]

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Treatment with 48% HF for a shorter period proved to be effective in creating deep micro-retentions on the topographic surface of zirconia in a more uniform manner when compared to that with sandblasting.[5] Recently, a mixed solution of HF and nitric acid has been introduced that was shown to create surface irregularities on the zirconia samples which were more uniform and detailed than those treated with sandblasting alone.[8] Only a few studies have noted the combination HNO_3/HF etching and it has shown that this combination significantly roughens the zirconia surface.

Cement selection is also an important prerequisite as the quality and longevity of its bond strength to zirconia ceramic profoundly determines the clinical success of the restoration. Resin cements can be classified as conventional resin cement and self-adhesive resin cement based on the bonding mechanism. Self-adhesive resin cements belong to a new category of resin cements that along with the ease of application, offer a few advantages of adhesive resin cements such as increased compressive strength and shear bond strength together with a good marginal seal.[9] These cements do not require technique-sensitive steps like etching, priming or bonding. G-Cem is a dual-cure adhesive resin cement, containing methacryloyloxydecyl dihydrogen phosphate (MDP) which shows excellent bonding to zirconia due to its chemical interaction between the functional phosphoric acid monomer and the hydroxyl groups of the passive zirconia surface. RelyX U200 is yet another dual cure self-etch resin cement which in addition to conventional methacrylate monomers contains methacrylate monomers with phosphoric acid groups as well as unsaturated carbon-carbon double bonds.

Luting zirconia is highly challenging as most surface treatments do not result in significant alteration of the surface due to the high crystalline content and limited vitreous phase of this ceramic. Though zirconia restorations can be luted using conventional cements, the choice of resin luting cements offers reliable bonds with higher bond strength. However further research targeting the adhesiveness of MDP-based resin cements to zirconia along with different zirconia surface pretreatment methods is necessary for product evaluation.

The objective of this study was to assess the effectiveness of various surface treatments along with different resin cements on the bond strength of zirconia-based ceramic to dentin.

Materials and Methods

In the present *in vitro* study, 80 caries-free sound permanent maxillary and mandibular premolars extracted for orthodontic purposes were selected.

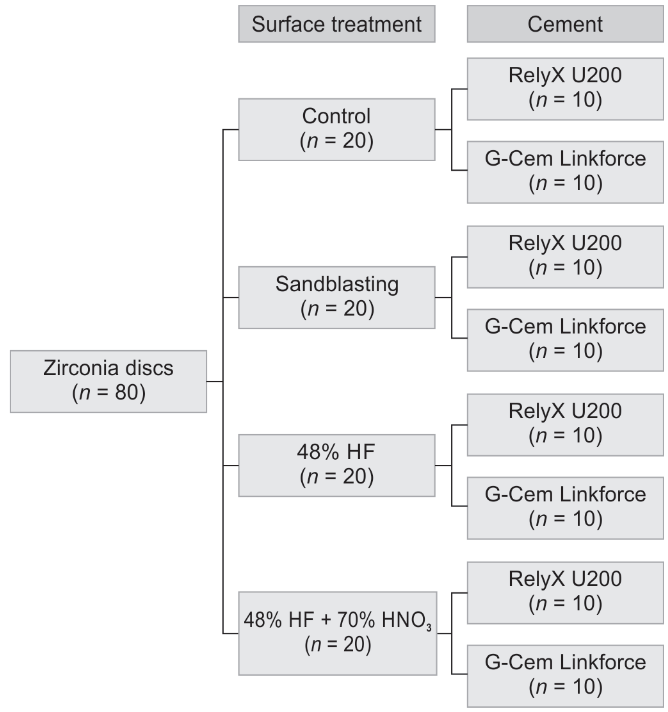
Tooth Preparation

The occlusal surface of the selected teeth was prepared to create a uniform dentinal surface. The prepared teeth were then embedded in an autopolymerizing resin such that the exposed dentinal surface was available for the cementation of zirconia samples until the bonding procedure.

Sample Preparation and Surface Treatment

A prototype of the Zirconia disc (Ceramill Zolid HT+, Denspro, Germany) was fabricated using a CAD/CAM system, measuring 3 mm in height and 4 mm in diameter. Using this prototype, 80 cylindrical samples of zirconia were milled from 4 blanks and were allotted into 4 main groups randomly (n = 20) based on surface treatment protocol (Flowchart 1).

Flowchart 1: Flowchart for different groups with subgroups



- Group I (G I): No surface treatment–control group.
- Group II (G II): Sandblasting was carried out with 50 µm aluminum oxide particles for 10 seconds at 2 bar pressure and at a distance of 10 mm.[10,11]
- Group III (G III): The samples were etched with 48% HF solution (NICE chemicals Pvt. Ltd, Kerala, India) with a micro applicator tip for 2 minutes and rinsed with deionized water for 1 minute and then gently air-dried.[5]
- Group IV (G IV): The samples were etched with 48% HF + 70% HNO_3 solution with a micro applicator tip for 2 minutes and were rinsed thoroughly with distilled water (NICE chemicals Pvt. Ltd, Kerala, India) and dried.[5]

The samples in each group were further subdivided into two subgroups with ten samples each based on the resin cement used for luting (Flowchart 1):

- A: RelyX U200 (3M, Germany): The cement was dispensed and mixed for 20 seconds and applied on the treated zirconia surface. The zirconia sample was cemented onto the exposed dentin surface. The cement was then light-cured on each side for 20 seconds according to the manufacturer's recommendations.
- B: G-Cem Linkforce (GC corporation, Tokyo): Total etch technique was adopted for surface treatment of tooth structure followed by application of the bonding agent (G-Premio bond, GC America Inc.) for 10 seconds, air dried for 5 seconds, and light cured for 10 seconds based on manufacturer's instructions. The zirconia samples were coated with the primer (G-Multi Primer, GC America Inc.) and dried. The cement was injected onto the cementing surface of zirconia which was seated over the treated tooth surface and light cured.

Following the bonding procedure, specimens were stored in distilled water at room temperature until testing within 24 hours.

Table 1: Mean ± SD of shear bond strength values of different study groups

Group (n = 10)	*Mean ± SD (MPa)*
Control–RelyX (GIA)	4.954 ± 0.972
Control G-Cem (GIB)	6.752 ± 0.740
Sandblasting RelyX (GIIA)	8.629 ± 0.850
Sandblasting G-Cem (GIIB)	12.996 ± 0.821
48% HF RelyX (GIIIA)	14.027 ± 0.845
48% HF G-Cem (GIIIB)	16.220 ± 1.574
48% HF + 70% HNO_3 –RelyX (GIVA)	12.389 ± 0.744
48% HF + 70% HNO_3 –G-Cem (GIVB)	11.281 ± 0.773

Assessment of Shear Bond Strength (SBS)

The samples were mounted onto a custom-made jig and subjected to shear stress using a Universal testing machine at a crosshead speed of 0.5 mm/minute until the debonding occurred. The force at which the samples de-bonded was expressed in Newtons (N). The resultant SBS was obtained in megapascals (MPa) by dividing the maximum force obtained in Newton by the cross-sectional area (mm^2) of the bonding surface for each specimen.

The formula used for the calculation was as follows:

- Area of the bonded surface (mm^2) = $\pi \times r^2$ (*r*–radius in mm).
- Shear bond strength (MPa) = Force (N)/Area of bonded surface (mm^2).

Statistical Analysis

Observed data was coded, tabulated, and analyzed using SPSS Version 20 for Windows. Shear bond strength was calculated according to the prescribed formula and expressed as mean and standard deviation. Since the data did not follow normal distribution, the values were represented as median and interquartile range and to test the statistical significance of the difference in bond strength among the four groups, Kruskal–Wallis ANOVA was applied. To compare the bond strength between two resin cements in each of the surface treatment groups, the Mann–Whitney *U* test was applied.

A *p*-value of less than 0.05 was considered statistically significant.

Results

A total of 80 samples (10 in each group) were analyzed. Table 1 shows the mean and standard deviation of shear bond strength of the study groups. The highest shear bond strength was noted for the group that used surface treatment—48% HF luted with G-Cem resin cement (16.220 ± 1.574) and the least for control–RelyX (4.954 ± 0.972). Luting with G-Cem showed higher mean bond strength for all surface treatments except for 48% HF + 70% HNO_3.

The null hypothesis was that the surface treatments of zirconia, with either sandblasting or acid etchants, followed by cementation with either self-adhesive resin or MDP containing self-etch resin, would not affect the bond strength of the zirconia to tooth structure when compared to the control group.

A comparison of median and IQR values between 8 groups revealed a statistically significant difference ($p < 0.001$) (Table 2 and Fig. 1). The highest mean SBS values were seen in 48% HF and cemented with G-Cem cement (15.70 IQR: 15.09–15.79) followed by

Table 2: Comparison of median shear bond strength between the study groups

Group (n = 10)	*Median*	*IQR*	*p-value*	*Order*
Control–RelyX (GIA)	5.083	4.325–5.746	<0.001*	8
Control–G-Cem (GIB)	6.792	6.051–7.482		7
Sandblasting–RelyX (GIIA)	8.941	7.812–9.335	<0.001*	6
Sandblasting–G–Cem (GIIB)	12.790	12.340–13.710		3
48% HF–RelyX (GIIIA)	13.857	13.511–14.556	0.002*	2
48% HF–G-Cem (GIIIB)	15.708	15.095–17.798		1
48% HF + 70% HNO_3 –RelyX (GIVA)	12.308	11.940–12.804	0.003*	4
48% HF + 70% HNO_3 –G-Cem (GIVB)	11.453	10.774–11.877		5
p-value < 0.001*				

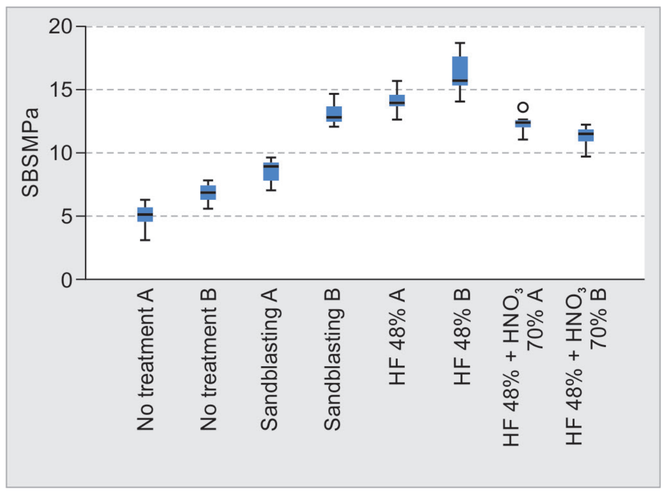


Fig. 1: Box plot-depiction of the shear bond strength values among the groups through their quartiles

48% HF–RelyX (GIIIA) (13.85, IQR: 13.51–14.55), sandblasting-G-Cem (GIIB) (12.79, IQR: 12.34–13.71), 48% HF + 70% HNO_3–RelyX (GIVA) (12.30, IQR: 11.94–12.80), 48% HF + 70% HNO_3–G-Cem (GIVB) (11.45, IQR: 10.77–11.87), sandblasting-RelyX (GIIA) (8.94, IQR: 7.81–9.33), control–G-cem (GIB) (6.79, IQR: 6.05–7.48) and control-RelyX (GIA) (5.08, IQR: 4.32–5.74).

A subgroup analysis was done based on the cement used for luting (Table 3). Thus, the highest bond strength was noted for 48% HF with G-Cem and the least for the Control group RelyX.

Discussion

In the recent decade, zirconia-based ceramics have gradually gained popularity as a promising biomaterial owing to the escalating

Table 3: Comparison of median shear bond strength between the subgroups (Resin cements)

Resin cement	*Surface treatment*	*Median (IQR)*	*p-value*
RelyX	Control	5.08 (4.32–5.74)	<0.001
	Sandblasting	8.94 (7.81–9.33)	
	48% HF	13.86 (13.51–14.55)	
	48% HF + 70% HNO_3	12.31 (11.94–12.80)	
G-Cem	Control	6.79 (6.04–7.48)	<0.001
	Sandblasting	12.79 (12.34–13.70)	
	48% HF	15.71 (15.09–17.79)	
	48% HF + 70% HNO_3	11.45 (10.77–11.88)	

demand for esthetics. However, the inertness of zirconia to traditional chemical and mechanical surface treatment methods that are applicable to glassy ceramics has compelled the researchers to explore an alternative strategy.[12]

Various studies have been carried out using several other strong acids such as nitric acid, sulfuric acid, and hydrochloric acid.[13] In the present study, the effects of sandblasting, HF etching alone and a combination of HNO_3/HF-etching on the zirconia surface were investigated by examining the shear bond strength between zirconia and adhesive resin cement. The current study employed the use of the G-Cem Linkforce system and RelyX U200 to maximize the bonding effectiveness of the zirconia.

Extracted natural teeth were used in the current study as they exhibit properties such as modulus of elasticity, thermal characteristics, strength and bonding features that are closer to the clinical scenario. Zirconia samples were milled using a prototype fabricated from CAD/CAM as this technology standardizes the design and fabrication of samples thereby delivering identical specimens for the *in vitro* studies of restoration materials, cements, or bonding techniques.[14]

The SBS values upon applying 48% HF solution were found to be more than that of the control group as it caused the dissolution of surface zirconia particles.[8] Furthermore, the SBS values of the mixed etching solution were merely more than the sandblasting group but less than that of the 48% HF group indicating reduced surface roughness created by the combined acid etching solution. Similarly, Liu et al. found that the surface roughness created by the combination of HF and HNO_3 acid solution was only 21% of that created by the HF acid.[15] Similar findings were also observed in a study by Goyata et al. wherein, a combination nitric acid and HF solution proved to be less effective in increasing the surface roughness of zirconia when compared to that of 48% HF alone.[5] The samples etched with HF alone and luted using G-Cem Linkforce exhibited the highest bond strength probably due to the presence of Zr-F bonds on HF-treated zirconia which improves the surface reactivity and due to the formation of fine and uniform nano-scale pits along with enlarged surface area when the zirconia was pretreated with HF. Such a shallow surface architecture advocates increased adhesion without forming deep and excessive surface damage that can weaken the treated zirconia. Quentin et al. demonstrated that a higher concentration of HF was required to etch the zirconia surface at room temperature, as it created an accelerated and highly uniform etch pattern.[16] The use of MDP-containing adhesive resin cement along with this HF pretreatment group would have also contributed to better bond strength values as functional monomers like MDP assure adhesion to enamel and dentin tissues, since the phosphoric acid and carboxylic acid functional groups of those monomers can chemically bond to calcium.

In the current study, G-Cem Linkforce showed higher bond strength values than the RelyX U200 resin cement which can be explained by its excellent hydrolytic stability owing to the presence of a functional monomer (MDP).[17] G-Multi Primer that was coated onto the zirconia surface before the application of G-Cem Linkforce resin cement, incorporates three main functional agents (silane, phosphate, and thiophosphate monomers), which provide adhesion to various substrates. The objective of adding silane to the primer is to enhance the stability of the bond.[6] The MDP monomers stimulate the formation of a chemical bond to the oxide group of zirconia *via* two hydrogen groups derived from the phosphate group forming a Zr-O-P covalent bond.[18,19] This Zr-O bond formation was confirmed by Byeon et al. proving the establishment of a chemical bond between zirconia and MDP monomer.[20]

However, the bond strength values of 48% HF + 70% HNO_3 treated samples cemented with G-Cem Linkforce luting agent (GIVB) were found to be less than that of those cemented with RelyX U200 cement (GIVA) probably because this self-adhesive resin cement percolates through spaces in the roughened surface to form microchemical interlocks and the inorganic filler in the cement is more resistant to hydrolytic degradation.[8] This finding is in concurrence with the results obtained by Oyagüe et al.[21]

The samples belonging to sandblasting surface treatment group displayed better bond strength values than that of the control group which could be because sandblasting with alumina particles generates a rough surface accompanied by a greater surface energy and wettability thereby enhancing the resin to zirconia bond The tetragonal-monoclinic (t-m) phase transformation generated by sandblasting of zirconia induces compressive stresses on the surface which counteracts the deleterious effects associated with it. However, the further progression of this phase transformation may develop tensile residual stresses thereby decreasing the flexural strength of the material. The effect of sandblasting on the flexural strength of zirconia is disputable with regard to the power and duration of the procedure.[22] In the present study, wherein the sandblasting group that was cemented with resin cement containing MDP monomer (G-Cem Linkforce) gave higher SBS values than the ones cemented with resin cement without the monomer (RelyX U200).[23]

Conclusion

Surface treatments based on HF etching, HF and HNO_3 combination significantly increased the bond strength of Zirconia compared to that observed in untreated specimens. The MDP molecule containing resin cement (G-Cem) showed better bond strength compared to self-adhesive resin cement. Further studies on strategies to enhance the adhesive strength of zirconia such as alteration in duration, concentration and temperature of the etchant are necessary. Additional *in vivo* trials will be needed to authenticate these *in vitro* results. Also, the effects of aging on Zirconia-resin bonding to mimic a clinical setting using these pre-treatment methods should be evaluated in further studies.

Ethical Approval

The review was approved by the Institutional Ethics Committee of Amrita Institute of Medical Sciences (IRB-AIMS-2019-161 dated 19-03-2019).

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