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AN INVITRO EVALUATION OF THE EFFECT OF ZIRCONIA SURFACE TREATMENT ON SHEAR BOND STRENGTH TO A RESIN CEMENT

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ABSTRACT: Statement Of Problem;Developments in ceramic core materials such as lithium disilicate, aluminium oxide, and zirconium oxide have allowed more widespread application of all-ceramic restorations over the past 10 years. However, establishing a reliable bond of resin cement to zirconia based materials has been proven to be difficult, which is the major limitation against fabricating zirconia restoration , and there is no clear recommendation in the literature regarding surface treatment before bonding. **Purpose;** This study evaluated the effect of zirconia surface treatments namely Airborne Particle Abrasion, Hydrofluoric acid etching, Hydrofluoric acid etching followed by silanation and Primer application on shear bond strength to a self etch dual cure resin cement bonded to dentin specimens. **Material And Methods;** Twenty zirconia rods (3 x 2.5 mm) were prepared from zirconia blocks and assigned into 5 groups. Each group were subjected to the following surface treatments. (1) group I - Control (C) no treatment , (2) group II - airborne-particle abrasion (APA), (3) group III – hydrofluoric acid etching (HF), (4) group IV - hydrofluoric acid etching followed by silanation (HF/S), and (5) group V - application of zirconia primer (Z) . Dentin specimens were prepared from extracted molars stored in 0.5% chloramine-T. Zirconia rods were bonded to dentin using a resin cement (Multilink Speed), then light polymerized. The specimens were loaded to failure with the notched shear bond test method in a universal loading apparatus. Results were analyzed using 1-way ANOVA (alpha=.05). **Results;**ANOVA showed significant differences in bond strength among the different surface treatments (p value 0.001). The highest values were obtained with group V (Z) (8.66 Mpa) followed by group II (APA) (6.71 Mpa), group IV (HF/S) (4.41 Mpa) . The least values were obtained for group III (HF) (3.88 Mpa) and there were no significant difference (p value 0.53) between group III (HF) (3.88 Mpa) and group I (C) (3.70 Mpa).**Conclusion:**The resin bond to Y-TZP was improved by surface treatment. Zirconia Primer application is practically a reliable surface treatment . Airborne Particle Abrasion , although not so reliable , is also an effective treatment while bonding zirconia to a resin cement .

KEYWORDS: Zirconia toughened alumina, Zirconia rod, selfcure resin

INTRODUCTION

Zirconia based ceramics are gaining popularity especially in fixed prosthodontics because of their superior mechanical properties and the development of new fabrication technologies for the all ceramic crowns and fixed partial dentures [17] . Zirconia based ceramics are recommended for FPDs , as they have the highest failure loads when compared to alumina and lithium di silicate based ceramics[6]. The commonly used Zirconia containing ceramic systems in recent times are Yttrium cation doped tetragonal Zirconia polycrystals (3Y-TZP), Magnesium cation doped partially stabilized Zirconia (Mg-PSZ), Zirconia toughened alumina (ZTA). Among the three, Yttria stabilized tetragonal zirconia (Y- TZP) has opened new vistas for all ceramic restorations. High flexural strength and fracture toughness afford its applications as framework material for FPD even in loaded reconstructions in the molar region[7]. Transformation toughened zirconia (Y – TZP) may stand out as the most successful all ceramic system, irrespective of the clinical situation, as the success rate for the posterior zirconia FPDs was 97.8 %.[2]

Heather [6] stated that the strength of an all ceramic restoration is dependent on the ceramic material used, core veneer bond strength , crown thickness , and design of the restoration, as well as bonding techniques and the characteristics of the supporting material. Previous investigations revealed that most clinical failures have initiated from the cementation or internal surfaces. Failure rates due to high strength ceramic fractures have been reported to range between 2.3 and 8 % . Therefore the integrity of the luting cement to ceramic surfaces plays a major role in the longevity of the restoration and the failures originated from cementation surfaces identified the

Fig.1 Zirconia rod

Fig.2 Tooth Specimens mounted on selfcure resin

Fig.3 Compression testing

need for a reliable conditioning method like surface treatment to strengthen this critical area [15]. The surface treatments available are of two types namely mechanical and chemical surface treatments. The mechanical treatments are dry or wet hand grinding , air particle abrasion and silicoating. The chemical treatments are HF acid etching, Silanation and primer application.

The objective of this study was to evaluate the effect of different sutface conditioning methods for zirconia on its bond strength to a resin cement

Materials and Methods

This study was done in Department of Dentistry , Indra Gandhi Medical College and Research Institute, Puducherry, India. This study evaluated the effect of zirconia surface treatments namely Airborne Particle Abrasion, Hydrofluoric acid etching, Hydrofluoric acid etching followed by silanation and Primer application on shear bond strength to a self etch dual cure resin cement bonded to dentin specimens.

Twenty Zirconia rods (3.6mmx3mm) were milled from Zirconia blocks (Incoris ZI, Sirona) using CAD/CAM (Sirona dental systems, GmbH) and sintered at 1500°C for 7 hours (Sintramat; Ivoclar Vivadent AG). Following sintering the rods measured approximately 3mm in diameter and 2.5mm in length (**Fig.1**).

One side of each rod was finished manually using 600 grit silicon carbide paper to make a flat surface. The rods were then divided into 5 groups of 4 rods in each group . Each group was mounted separately in a light body vinyl polysiloxane material (Zeta plus; Zhermack) and subjected to the following surface treatments.

- The group I (C) was the control group where no surface treatment was done.
- The group II (APA) zirconia rods were subjected to Airborne particle abrasion with 50µm Al_2O_3 particles at 3.0 bar pressure from a distance of 10mm parallel and perpendicular to the long axis of the bars, for 10 seconds.
- The group III (HF) zirconia rods were subjected to hydrofluoric acid 4.5% etching (IPS Ceramic etching gel; Ivoclar Vivadent AG) for 3 minutes and then dried.
- The group IV (HF/S) zirconia rods were subjected to hydrofluoric acid 4.5% etching (IPS Ceramic etching gel; Ivoclar Vivadent AG) for 3 minutes and silanated by application of a silane coupling agent (Monobond-S; Ivoclar Vivadent AG) for 2 minutes, then dried.
- The group V (Z) zirconia rods were subjected to application of Zirconia primer ((Metal/Zirconia primer; Ivoclar Vivadent AG) on intaglio surfaces of the rods for 3minutes,then dried. All applications (HF, S, Z) were done using application brushes.

Twenty extracted permanent molars were collected and stored in 0.5% chloramine T (Explicit Chemicals Pvt Ltd, Pune) for 1 week. The buccal and lingual surfaces of each molar were ground flat using 600-grit Silicon carbide paper to expose dentin. Each tooth was sectioned longitudinally in a mesiodistal direction using a precision saw with a high concentration diamond watering blade underwater cooling. Tooth sections were mounted in autopolymerizing acrylic resin (DPI Cold cure) using molds measuring 25mm in diameter and 26mm in height (**Fig.2**). The molds were immersed in cold water during polymerization of the acrylic resin to prevent overheating of the specimens. Specimens were recovered, ground flat on both sides using a model trimmer, and stored in tap water, then assigned into 5 groups (n=4).

Each group was assigned one of the pretreated Zirconia rod groups. The exposed dentin surface was finished with 600grit 8 inch grinding disks, rinsed with water, and gently air dried. A dual polymerizing, self etching adhesive cement (Multilink Speed, Ivoclar Vivadent AG) was used to bond the Zirconia rods to dentin specimens according to manufacturer's instructions. Specimens were placed in a bonding clamp and tightened with light finger pressure . Excess cement was removed, and the margins were light polymerized using a halogen polymerization light (3M ESPE) at 1350mw/cm^2 for 20 seconds on both sides of the specimens . The specimens were stressed with notched shear bond test method in a universal loading apparatus (Unitek 94100, FIE) setup for compression testing using a .05-kN load cell at 1mm/min cross head speed (**Fig.3**). All specimens were prepared and tested by the same operator to eliminate intero operator variability. One way ANOVA followed by Tukey HSD for cell means were used to analyze the data (α=.05), with mechanical and chemical treatments as independent variables.

Results

All specimens showed failure at zirconia resin interface. ANOVA showed significant differences in bond strength among the means with different surface treatments with the p value 0.001 . The highest values were obtained with group V (Z) (8.66 Mpa) followed by group II (APA)(6.71 Mpa). The least values were obtained for group I (C) (3.70 Mpa) . There were no significant difference (p value 0.53) between group III (HF) (3.88 Mpa) and group I (C) **(Table-1) (Graph-I)**

It is inferred from the result that there is a statistically significant difference among the groups (P<0.01). The mean value of group V (Z) is the highest among other groups.

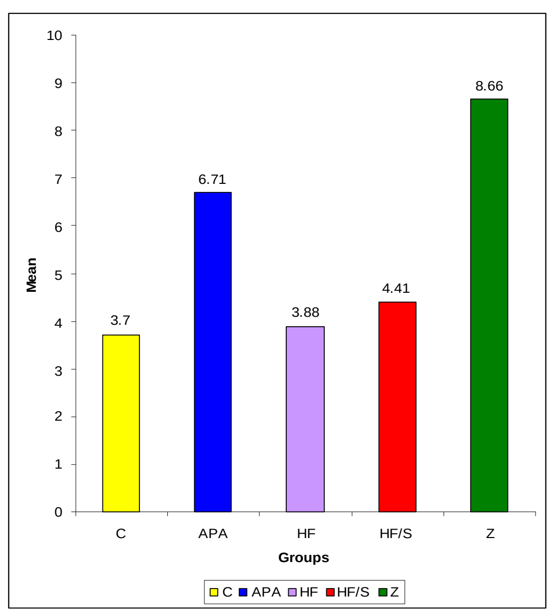
Table1: **Mean, Standard Deviation and F-values for all groups**

Group	N	Mean	SD	F-value	P-value
C	4	3.70	0.35	129.52	0.001 Significant
APA	4	6.71	0.40		
HF	4	3.88	0.40		
HF/S	4	4.41	0.35		
Z	4	8.66	0.35		

Discussion

In this study, Zirconia rods (InCoris ZI, Sirona) were surface treated using group I - control (C) no treatment , (2) group II - airborne-particle abrasion with 50 µm Al_2O_3 (APA), (3) group III - hydrofluoric acid etching (4.5%) etching with IPS Ceramic etching gel (Ivoclar vivadent) (HF), (4) group IV - hydrofluoric acid etching followed by silanation with a silane coupling agent Monobond S (Ivoclar Vivadent) (HF/S), and (5) group V - Metal / Zirconia primer (Ivoclar Vivadent) application (Z). Then they were luted to dentin specimens with a dual cure self etching resin cement (Multilink Speed, Ivoclar Vivadent) and then subjected for shear bond strength test using a Universal testing machine (Unitek 94100, FIE).

In this study, the comparison of surface treatments for shear bond strength were statistically analyzed and the p value was significant (0.001) (Table 1) The group I (C) was the least with a mean of 3.70 Mpa. The group V (Z) achieved the maximum bond strength of 8.66 Mpa .The group II (APA) achieved 6.71 Mpa . The group IV (HF / S) attained the bond strength value of 4.41 Mpa .



Graph.1 Bar diagram showing Mean values for all groups

The group III (HF) attained the least bond strength value (3.88 Mpa) among surface treatment groups. Also there was no significant difference with the control group . Etching the inner surfaces of ceramics with glassy matrix using Hydrofluoric acid followed by the application of silane coupling agent is an efficient conditioning method for bonding resin composite.[15] **Torres**[16] evaluated the effect of acid ectching on zirconia by SEM analysis and reported that the surface morphology was not changed by HF acid etching. This was because the use of hydrofluoric acid selectively dissolves the glassy components of silica based ceramics, producing a porous, irregular surface areas and facilitates the penetration of the resin cement. This chemical reaction was not applicable to zirconia based ceramics because of the lack of a silica phase . Hydrofluoric acid etching was an inadequate surface treatment for bonding resin to zirconia ceramic[5].

The group IV (HF /S) attained the bond strength value of 4.41 Mpa ranking just above HF group. Achieving a strong and durable bond to glass ceramic depends on applying HF followed by application of silane coupling agent [9].HF acid attacks the glass phase producing a retentive surface [11] suitable for micromechanical bonding, and the silane coupling agent promotes a chemical bond between the silica phase of these ceramics and the methacrylate groups of the silane coupling agent. On the other hand, this method was claimed not effective for the glass free zirconium oxide ceramics used in this study as its composition and physical properties makes this material resistant to the acidic or alkaline corrosive materials [13]. In a study by **Derand** [3], Silane treatment reduced the bond strength and it was concluded that as expected the surface did not react with silanes at room temperature . However, Silanes have a greater capacity to wet the surface[1]. This improved wetting ability of the surface may have resulted in a small but increased bond strength value.[3] This may be the reason why the HF/S group has slight more value than HF group although both HF and S treatment did not produce a chemical bond, the wetting ability of the Silane coupling agent has improved the bond strength, though in a small amount.

The group II (APA) achieved 6.71 Mpa ranking second , following the Z group. This was in agreement with a study by **Qublewi**[14] where the APA specimens ranked second next to group V (Z), leaving behind Hydrochloric acid etching and silanation and control groups. APA not only increased the bond strength to resin but also the flexural strength of the zirconia[14, 8]. APA is a prerequisite for achieving bond strength between the resins and high strength ceramics that are reinforced either with alumina or zirconia. The APA process removes loose contaminated layers and the roughened surface provides some degree of mechanical interlocking or keyingwith the adhesive. It can be due to the fact that the increased roughness has also increased the surface area for the bond [15]

The group V (Z) achieved the maximum bond strength of 8.66 Mpa ranking above all. This is agreement with the study by **Qublewi** [14], **Aboushelib**[12], **Esam** [4]. Metal/Zirconia Primer is a single-component priming agent designed to mediate an optimal chemical bond between metal alloys or oxide ceramics (zirconium oxide, aluminium oxide) and methacrylate-based luting composites. Metal / Zironia Primer contains a phosphoric acid compound as the active ingredient, which establishes a chemical bond to oxidic surfaces. These phosphate monomers are very effective in improving cohesive zirconia bonding to resin cements. This reliable bond is critical especially in a minimally retentive case such as veneers [10].

The limitations of this study are the specimens were not subjected Artificial aging / Thermocycling. Static loading was used instead of dynamic loading, which is more common in intra oral conditions. The result of this study was based on the use of a resin cement supplied by a particular manufacturer which cannot be generalized for other resin cements.

However within the limitations of this study, it is conclusive of the fact that the Metal/ Zirconia primer application is the best surface treatment for zirconia ceramics followed by Air Particle Abrasion with 50 µm Al_2O_3 particles among the other surface treatments in this study.

CONCLUSION

Within the limitations of this invitro study it is conclusive of the facts that

1. Conditioning of Y-TZP can improve its bond to the resin cement
2. Zirconia primer application is a practical procedure resulting in improved bond strength followed by Airborne Particle Abrasion

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