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Combination of Selective Infiltration Etching Along with Acid Etching and KHF2 Etching on the Bond Strength between Zirconia and Ceramic: An Invitro-Study

Dr. Kiran Kumar N

Professor and Head of the Department, Department of Conservative Dentistry & Endodontics, Government Dental College and Research Institute, Fort, Bangalore – 560002, India .

Dr. Anoop Nair

Department of Prosthodontics and Implantology, Government Dental College and Research Institute, Fort, Bangalore – 560002, India.

Dr. Hariprasad L*

Post graduate student, Department of Conservative Dentistry & Endodontics, Government Dental College and Research Institute, Fort, Bangalore – 560002, India.

Dr. Priya Mariam Thomas

Post graduate student, Department of Conservative Dentistry & Endodontics, Government Dental College and Research Institute, Fort, Bangalore – 560002, India.

Dr. Biji Brigit

Associate Professor, Department of Conservative Dentistry & Endodontics, Government Dental College and Research Institute, Fort, Bangalore – 560002, India.

Dr Savitha B Naik

Associate Professor, Department of Conservative Dentistry & Endodontics, Government Dental College and Research Institute, Fort, Bangalore – 560002, India.

ABSTRACT

Etching of zirconia is commonly employed to enhance the bond between zirconia and ceramic. A combination of various etching procedures are employed to enhance the bond strength. A 150 cuboid shaped zirconia samples were employed for the study and were divided into 5 groups. Group 1: sandblasting with 50microns Al_2O particles. Group 2: Hydrofluoric acid etching(HF). Group 3: KHF_2 etching. Group 4: HF + Selective Infiltration Etching (SIE). Group 5: KHF2 +SIE. Layering of the specimens were done with ceramic and the specimens were subjected to shear bond strength testing. Data was subjected to a normalcy test: Shapiro-Wilk test. Data showed non normal distribution. Hence non-parametric tests (Kruskal-wallis test with post hoc Mann-whitney) were applied. The SIE+HF group showed the maximum bond strength and lowest bond strength noticed in KHF2 etching group. The

*** *(Corresponding Author. Dr. Hariprasad L)*
E-mail Id: hariprasadl6789@gmail.com

sandblasted groups performed better than the KHF2 etching group. The study concluded that combination of various etching procedures in turn increased the bond strength than the surface treatment performed alone.

INTRODUCTION

Zirconium dioxide (zirconia) has become more widely used for ceramic restorations and in clinical practice. Given its outstanding mechanical properties, chemical stability, biocompatibility, and aesthetics, zirconia is now widely used in restorations, fixed dental prostheses, and superstructures of implant-supported prosthesis [1,2]

The opaque nature of zirconia compels for the usage of double-layered restoration i.e zirconia framework followed by veneering ceramic that produces an aesthetic color reproduction of natural teeth. [3] The major challenge that we face in such restoration is the property of the chemical stability of zirconia. The excellent bio inertness of zirconia causes the lack of adhesive bonding between the core and the veneering material.[4] This results in chipping or cracking of veneering ceramic leading to the failure of the restoration.[5]

Various surface treatments have been employed to enhance the bonding between the zirconia core and veneering ceramic. These surface treatments may increase the surface roughness or surface area thereby providing better adaptability and bond strength. Commonly used surface treatments to enhance bonding are sandblasting with aluminum oxide particles, acid etching, laser etching, liner application or a combination of any of the methods.[6]

One among the most significant protocols is the Selective infiltration etching as patented by Aboushelib et al in 2007. [7] This novel surface roughening method basically involves the infiltration of low fusing glass or organic oxides into the zirconia surface with heat induced maturation. Park et al[8] used the selective infiltration etching (SIE) method which employs conventional acid etching of zirconia using HF to create microporosities on the zirconia surface. SIE is associated with zirconia nanoparticles and glass conditioners to increase the bond strength. Various combinations(wt%) of zirconia nanoparticles and glass conditioner could be employed. They however advocated the usage of higher concentration of glass powder in the mixture of zirconia nanoparticles and glass powder.

This study aimed to evaluate the effect of SIE along with a combination of acid etching with HF or KHF_2 in the bond strength of zirconia to ceramic. The proposed hypothesis was etching with KHF2 or HF followed by SIE increases the bond strength.

MATERIALS AND METHODS

A hundred and fifty cuboid shaped zirconia blocks (5mm X 5mm X 10mm) CAD-CAM milled from a standard yttria stabilized zirconia blank (Amann Girrbach, Ceramill zi white, Singapore, Asia) were selected for this study. In order to obtain a standardized surface roughness, the blocks were polished with the 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, under water cooling. Further the blocks were divided into 5 groups, 30 in each group.

GROUP 1: Sandblasting: Al_2O_3 airborne-particle abrasion was performed using a sandblast machine with 50µm particle size for 15 seconds at a 10mm distance from the surface and with a pressure of 1.5 bar. Finally, the specimens were ultrasonically cleaned in 96% isopropyl alcohol for 3 minutes and steam-cleaned for 15 seconds.

GROUP 2: Hydrofluoric acid etching: 30 zirconia specimens were immersed in 9.5% hydrofluoric acid for at 25 degree Celsius for 1 hr.

GROUP 3: KHF_2 etching: 30 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 degree Celsius. The bonding surface was then cleaned for 15 secs using a steam cleaner for 15 secs followed by compressed air for 15 secs

GROUP 4: HF +SIE: 30 zirconia specimens were immersed in 9.5 % HF at 25 C for 1 hr. Followed by SIE.

GROUP 5: KHF2 +SIE: 30 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 degree Celsius. The bonding surface was then cleaned for 15 secs using a steam cleaner for 15 secs followed by compressed air for 15 secs. Followed by SIE.

The surface morphology was observed by a scanning electron microscope (SEM). Image of each surface will be registered at 2000x magnification.

LAYERING OF VENEERING 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 is condensed using the vibration blotting technique, fired and finally a glaze firing were performed according to the manufacturer's instructions. Each specimen was embedded at the centre of a metal holder, with the core-veneer interface positioned at the top level of the holder. 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 dwell time Then, these metal holders were mounted in a universal testing machine. Specimens were tightened and stabilized to ensure that the 1mm 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 were applied at a crosshead speed of 0.5 mm/min until fracture occurred. The ultimate load to failure were recorded in Newton (N). The average SBS (MPa) were calculated by dividing the load (N) at which failure occurred by the bonding area (mm^2) as follows:

$$\text{Shear stress (MPa)} = \text{Load (N)/surface area}$$

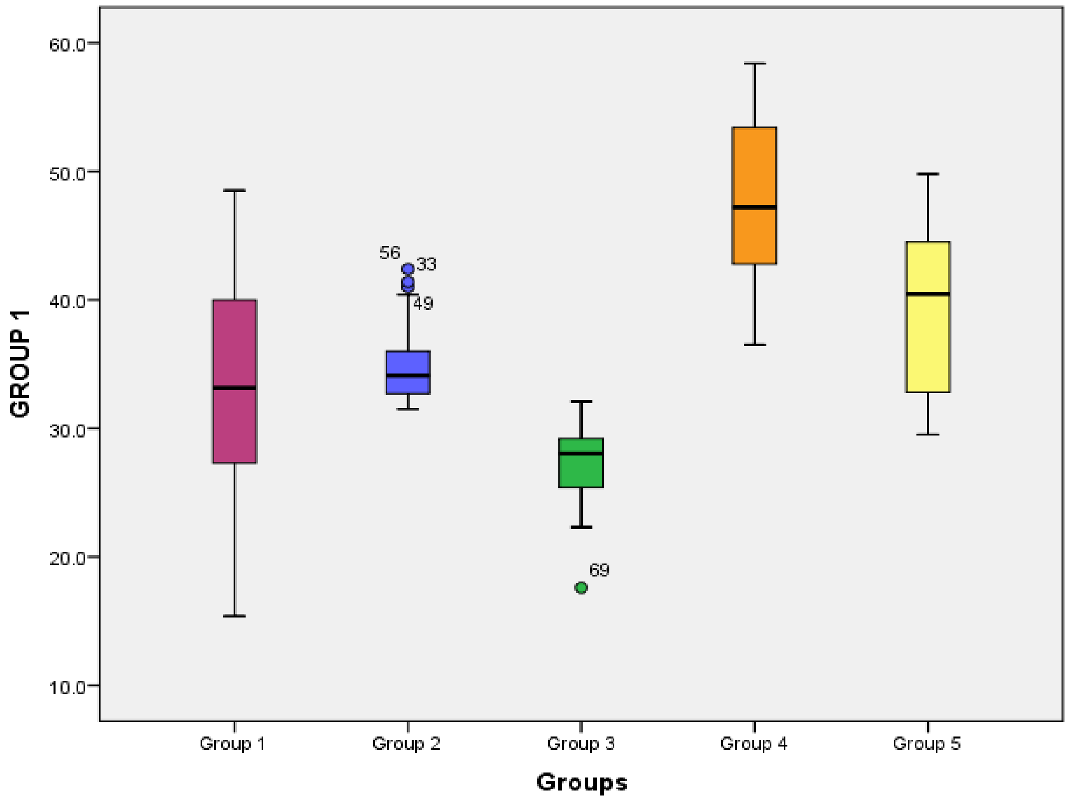
The fractured surfaces were visually analyzed with a stereomicroscope to determine the failure modes of specimens. Failure modes were classified as follows: cohesive fracture within the veneer, adhesive fracture between the core and veneer, or a combination of both.

DATA ANALYSIS: Data was subjected to a normalcy test: Shapiro-Wilk test. Data showed non normal distribution. Hence non-parametric tests (Kruskal-wallis test with post hoc Mann-whitney) were applied.

RESULTS

Groups	Minimum	Maximum	Median	IQR
Group 1	15.4	48.5	33.15	13.1
Group 2	31.5	42.4	34.10	3.5
Group 3	17.6	32.1	28.05	3.9
Group 4	36.5	58.4	47.20	10.9
Group 5	29.5	49.8	40.45	12.0

Shear bond strength was higher in Group 4- 47.20(IQR 10.9) followed by Group 5- 40.45(IQR-12), Group 2- 34.1(IQR- 3.5), Group 1- 33.15(IQR- 13.1) and Group 3- 28.05(IQR-3.9).



*p value set significant at 0.05/5=0.01

Inter group comparison was done using post-hoc Mann Whitney test. Statistically significant difference was seen between all the groups except between Group 1 v/s Group 2(p=0.371).

DISCUSSION

Zirconia material was introduced into the dentistry due to its superior properties such as high mechanical strength and biocompatibility. This material has been routinely used as a core material for the fabrication of crowns and long span bridges. Zirconia due to its better aesthetics and opaque nature is an excellent alternative to metal cores that were routinely used [9]. Zirconia-ceramic when subjected to loading causes the chipping off the veneering ceramic causing the failure of the restoration [10-14].

Various studies concluded that Hydrofluoric acid [HF] cannot be used to etch zirconia due to its high crystalline phase content. [15,16] Also several recent studies have demonstrated that HF can cause zirconia etching with phase change from tetragonal to monoclinic. Fluoride compounds when melted on zirconia surface produces a rough etched surface that can enhance adhesion. This study was done to evaluate the effect of etching of zirconia surface with HF or KHF_2 followed by selective infiltration etching SIE/HIM on the shear bond strength of zirconia to veneering ceramic. [7,17]

Based on the results obtained the null hypothesis was rejected. The specimens that were etched with HF followed by SIE showed significant increase in bond strength values. The zirconia specimens when treated with HF showed superficial grain anomalies. This was evident from the higher magnification SEM images which demonstrated change in grain shape, displacement of the grains superficially thereby increasing the inter-grain space. 9.5% HF can etch zirconia surface when treated for 1 hour at 25^0C or 9.5% HF for 1min at 80^0C. [18] The main effect of HF is by corroding the surface of zirconia thereby creating surface defects. Images revealed that the inter-grain distance increased at a nanoscale level. SEM images of HF treated groups showed corroded surface or surface defects on the zirconia. The surface was quite uneven with shallow valleys and hills noticeable on the surface. (Image 2). Etching with HF is non-controllable and this may have attributed to this change on the zirconia surface.

When these specimens were further treated with SIE/HIM it enhanced the penetration of the infitrant agent thereby creating changes ultra structurally. This may have attributed to the enhanced bond strength of zirconia to veneering ceramic. During SIE/HIM procedure temperature kept is between 750^0C and 650^0C. This further causes the grain boundaries of zirconia to contract and expand. The infiltrant agent when applied on the zirconia surface flows into the pre strained grain boundaries. The dissolution of this agent in HF gives the desired architecture for the zirconia surface. The three dimensional retentive areas created by SIE and the surface roughness by HF etching helps in the mechanical interlocking of ceramic to zirconia. SIE treated groups revealed similar surface characteristics. There were no larger surface defects with even surface morphology throughout. (Image 3 and 4). The noticeable difference in bond strength in group 4 and group 5 may be attributed to SIE rather than the initial etching procedure performed. The infiltrant used for SIE belongs to low fusing ceramic family to have a matching coefficient of thermal expansion to zirconia. This infiltrant is squeezed into the surface grains ultrastructural creating retentive areas on a nanoscale.[7]

KHF_2 etching has been recently introduced as a surface treatment of zirconia. Ruyter et al demonstrated that this fluoride compound when melted over zirconia produced etching of its surface layer. The efficiency of the etching is based on dissolution of ZrO2 in the molten K[F··H··F] and the affinity of F for Zr and the evaporation of water. Melt etching of zirconia with the fluorides created a rough surface of exposed zirconia grains with deep groves. Moreover, specimens etched with difluorides appear to have similar morphology to that of specimens etched with hydrofluoric acid. The KHF_2 group showed similar increase in bond strength when combined with SIE/HIM.

CONCLUSION

Frequently etching procedures are performed alone to created surface roughness on the zirconia surface. This is often not effective method, however the results of this study shows that combination of SIE and HF/KHF2 etching procedures significantly increased the bond strength with minimal loss of the zirconia surface layer. Further studies are required to elucidate the effect of these combination of surface treatment on zirconia.

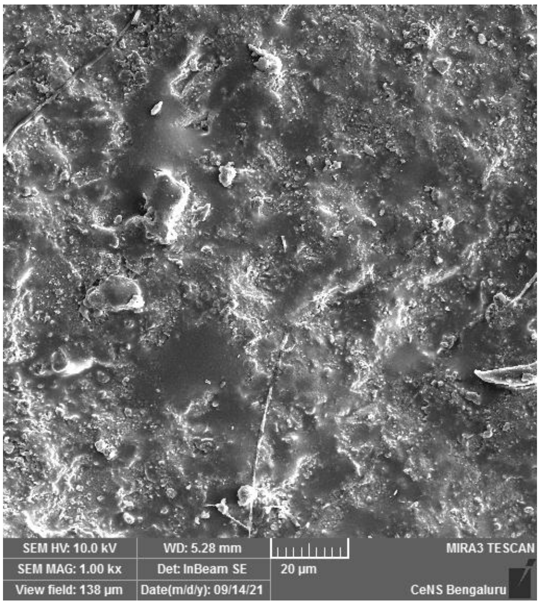


IMAGE 2

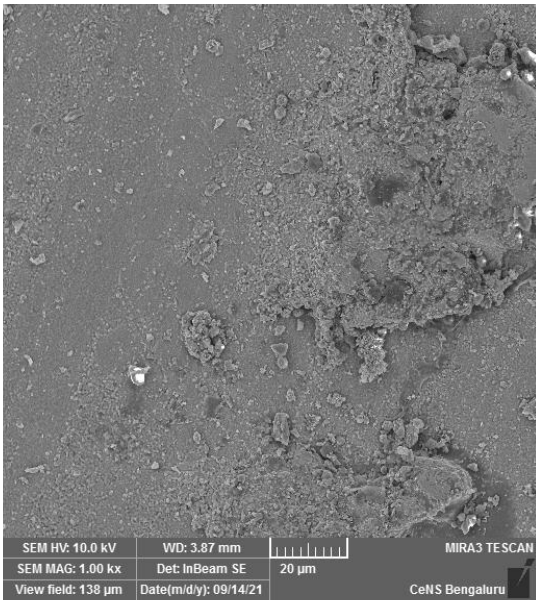


IMAGE 3

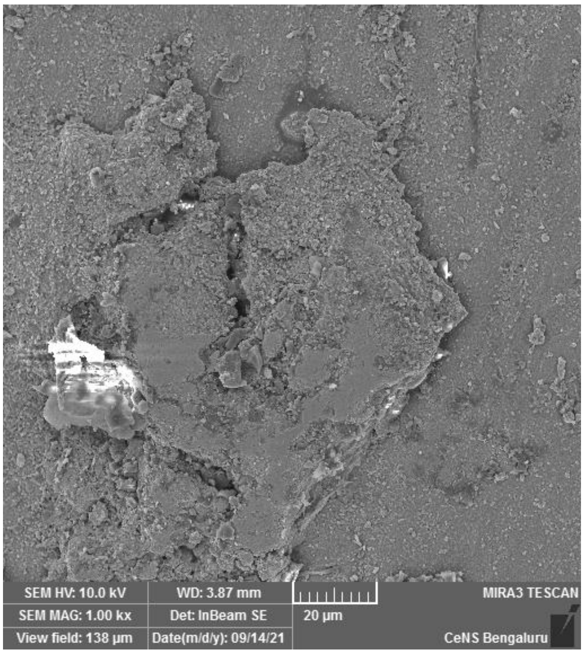


IMAGE 4

REFERENCES

[1] Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. J Prosthodont Res 2013; 57: 236-261.

[2] Bona AD, Pecho OE, Alessandretti R. Zirconia as a dental biomaterial. Materials (Basel) 2015; 8: 4978-4991.

[3] Park DR, Uhm SH, Kim KN, Bae JM, Kim JM, Jung SH, Kim JH, Oh S. The effect of selective infiltration etching (SIE) associated with zirconia nanoparticles on the shear bond strength of zirconia core/veneering ceramic in dentistry. J Nanosci. 2016;16(11):11957-63.

[4] Ansari S, Jahedmanesh N, Cascione D, et al. Effects of an etching solution on the adhesive properties and surface microhardness of zirconia dental ceramics. J Prosthet Dent. 2018;120(3):447-453. doi:10.1016/j.prosdent.2017.11.026

[5] Nishigori A, Yoshida T, Bottino MC, Platt JA. Influence of zirconia surface treatment on veneering porcelain shear bond strength after cyclic loading. J Prosthet Dent. 2014;112(6):1392-8.

[6] Mello CC, Bitencourt SB, dos Santos DM, Pesqueira AA, Pellizzer EP, Goiato MC. The effect of surface treatment on shear bond strength between Y-TZP and veneer ceramic: a systematic review and meta-analysis.J Prosthodont. 2018;27(7):624-35.

[7] Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials. J Prosthet Dent. 2007 Nov;98(5):379-88. doi: 10.1016/S0022-

3913(07)60123-1. PMID: 18021827.

[8] Park DR, Uhm SH, Kim KN, Bae JM, Kim JM, Jung SH, Kim JH, Oh S. The effect of selective infiltration etching (SIE) associated with zirconia nanoparticles on the shear bond strength of zirconia core/veneering ceramic in dentistry. J Nanosci. 2016;16(11):11957-63.

[9] Ruyter EI, Vajeeston N, Knarvang T, Kvam K. A novel etching technique for surface treatment of zirconia ceramics to improve adhesion of resin-based luting cements. Acta Biomater Odontol Scand. 2017 Apr 14;3(1):36-46. doi: 10.1080/23337931.2017.1309658. PMID: 28642930; PMCID: PMC5463337.

[10] I. Sailer, A. Feher, F. Filser, H. Luthy, L.J. Gauckler, P. Scharer, C.H.F. Hammerle, Prospective clinical study of zirconia posterior fixed partial dentures: 3-year followup, Quintessence Int 37(9)(2006) (685–693) (2006).

[11] M.F. Sola-Ruiz, R. Agustin-Panadero, A. Fons-Font, C. Labaig-Rued, A prospective evaluation of zirconia anterior partial fixed dental prostheses: clinical results after seven years, J. Prosthet. Dent. 113 (6) (2015) 578–584

[12] A. Ortorp, M.L. Kihl, G.E. Carlsson, A 3- year retrospective and clinical follow-up study of zirconia single crowns performed in a private practice, J. Dent. 37 (9) (2009) 731–736.

[13] A.J. Raigrodski, G.J. Chiche, N. Potiket, J.L. Hochstedler, S.E. Mohamed, S. Billiot, The efficacy of posterior three-unit zirconium-oxide-based ceramic fixed partial dental prostheses: a prospective clinical pilot study, J. Prosthet. Dent. 96 (2006) 237–244.

[14] C. Sax, C.H. Hammerle, I. Sailer, 10-year clinical outcomes of fixed dental prostheses with zirconia frameworks, Int. J. Comput. Dent. 14 (3) (2011) 183–202.

[15] Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/ cementation to zirconia and other non-silicate ceramics: where are we now? Dent Mater 2011; 27: 71-82

[16] Yun JY, Ha SR, Lee JB, Kim SH. Effect of sandblasting and various metal primers on the shear bond strength of resin cement to Y-TZP ceramic. Dent Mater 2010; 26: 650-658.

[17] Jevnikar P, Krnel K, Kocjan A, Funduk N, Kosmac T. The effect of nano-structured alumina coating on resin-bond strength to zirconia ceramics. Dent Mater 2010; 26: 688-696.

[18] Sriamporn T, Thamrongananskul N, Busabok C, Poolthong S, Uo M, Tagami J. Dental zirconia can be etched by hydrofluoric acid. Dent Mater J. 2014;33(1):79-85. doi: 10.4012/dmj.2013-243. PMID: 24492116.