DOI: 10.21276/aimdr.2020.6.5.DE2

Original Article ISSN (O):2395-2822; ISSN (P):2395-2814

Effect of 37% Phosphoric Acid Etchant on Surface Topography of Zirconia Crowns – An In Vitro Study

Adil Fayaz[1], Sandeep Kaur Bali[2], Mushtaq Mohammad Bhat[3], Aamir Bashir[1]

[1]Post-Graduate Student, Department of Prosthodontics, Govt. Dental College & Hospital, Srinagar, Jammu & Kashmir, India.
[2]Professor and Head, Department of Prosthodontics, Govt. Dental College & Hospital, Srinagar, Jammu & Kashmir, India.
[3]Endodontist, Health services, Jammu and Kashmir, India.

Received: July 2020
Accepted: July 2020

ABSTRACT

Background: This in vitro study was conducted to evaluate the surface topography of dental zirconia specimen after etching with 37% phosphoric acid. **Methods:** These samples were divided into 4 groups based on the etching time – with Group-I as a control and Group-II, Group-III and Group IV with etchant application time of 5, 10 and 15 minutes respectively at room temperature. **Results:** The specimens were evaluated under Scanning electron microscope. The SEM analysis revealed changes in surface topography for all the etched zirconia specimens. **Conclusions:** The micro-irregularities on the surface increased with longer etching times.

Keywords: Acid etching, Phosphoric acid, Zirconia, Scanning electron microscopy.

1

INTRODUCTION

Zirconia-ceramics is one of the most popular non-metallic material used in prosthetic dentistry owing to its superior mechanical properties, excellent esthetics & biocompatibility. Light transmission is approximately 48%, and the refractive index is 2.3.[1,2] Sintered yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) are described as 'polycrystalline, high-strength oxide ceramics' without a 'glass phase'.[3] However, the hardness of yttria stabilized zirconia (3Y-TZP) causes problems with surface enhancement and makes it difficult to ensure appropriate preparation for bonding between the luting cement and the prosthetic restoration.

The homogenous, densely packed structure of zirconia crystals and the absence of a glass matrix make etching and silanization impossible. For this reason, bond strength is lower than for glass ceramic restorations.[4,5] The minimum tensile bond strength acceptable in clinical conditions ranges between 10 and 13 MPa.[6] A strong, durable resin-to-ceramic bond is established through the formation of chemical bonds and by micromechanical interlocking.[7-9]

Ceramics based on silicates like dental porcelain and glass ceramics can be etched by hydrofluoric acid (HF in water) followed by silanization with an organosilane to achieve good bonding. Since zirconia is a silica-free ceramic and so resistant to conventional acid-etching techniques.

Name & Address of Corresponding Author
Dr. Adil Fayaz,
Gulshan Nagar, Nowgam,
By-pass, Srinagar,
Jammu & Kashmir, India.
Pin code: 190015.
Email: adifay786@gmail.com

Zirconia ceramic is not readily etched by HF owing to its high crystallinity, making it difficult to roughen the surface for mechanical retention.[2,10-14] On the other hand, air-abrasion using Al_2O_3 particles has been found to be effective in cleaning and roughening surfaces.[15,16] Wegner and Kern demonstrated that a durable resin–zirconia bonding can be achieved by air-abrasion in combination with resin materials containing an adhesive monomer, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP).[17] Other surface treatments of zirconia

including laser treatment selective infiltration technique (SIE) hot etching solution nanostructured alumina coating, and slurry-coated ceramic have been developed to improve and enhance the surface roughened area to facilitate the mechanical interlocking of the ceramic and bonding resin.[14,18-23]

The purpose of this study was to investigate the effect of 37% phosphoric acid, a commonly used etchant, on the surface topography of zirconia to examine whether surface roughness is affected or not by the increased etching times, which is essential for the interlocking of resin cement.

MATERIALS & METHODS

A total of 60 samples of monolithic Zirconia crowns designed for mandibular first molar each fabricated using CAD-CAM technology was used in this study. The samples were then cleaned for five minutes with acetone, ethanol and distilled water in an ultrasonic bath in order to remove contaminants to facilitate the surface treatment.

These samples were divided into 4 groups (n=15) based on the etching time. With one group (Group-I) as a control, specimens in other 3 groups were etched using 37% phosphoric acid. Etchant was applied to each of the 15 specimens in Group-II, Group-III and Group IV for 5, 10 and 15 minutes respectively. The volume of etchant for each sample was 1mL. After etching, the samples were rinsed off with acetone & cleaned in distilled water bath ultrasonically. The specimens were examined with a Hitachi S – 3000H scanning electron microscope for surface topography analysis.

The zirconia specimen crowns from each etched group were air-dried, mounted on a brass stub with carbon adhesive tape, and gold sputter coated for 3 min. Surface topography areas were randomly selected and photographed using an electron microscope at 500µ, 1000µ, 2000µ and 3000µ magnification.

RESULTS

Group –I

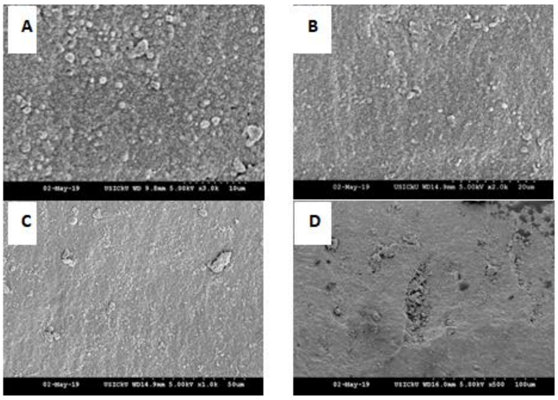

Figure 1: SEM image of surface of untreated specimen at (A)×500, (B) x1000, (C) x2000 and (D) x3000 magnification.

Group-II

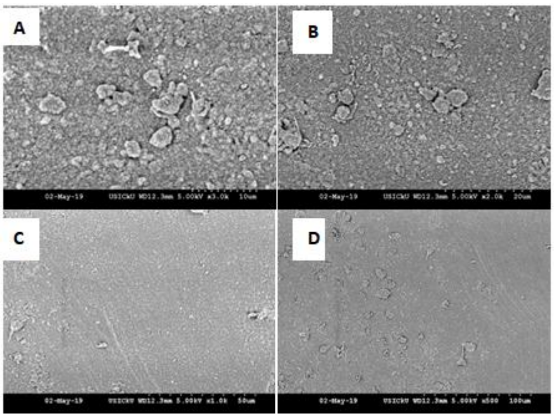

Figure 2: SEM image of surface of specimen after etching with 37% phosphoric acid concentrations after 5 minutes at (A)×500, (B) x1000, (C) x2000 and (D) x3000 magnification.

The unetched zirconia surface of the control specimens GROUP-I showed a homogenous fine grain structure. GROUP-II showed the specimen surface with an irregular grain structure compared with the control. Group-III specimens had the irregular grain shape and enlargement of inter-grain space. Group-IV images of the zirconia specimens revealed irregular morphology and formation of the micro-porosities more evident at higher magnification.

The scanning electronic microscopic images showed that the etchant effect was both at the inter-granular and intra-granular level with more preferentiality for the grain boundaries depending on etching time. Nevertheless, observations at higher magnification showed no significant evolution of the granular texture.

Group-III

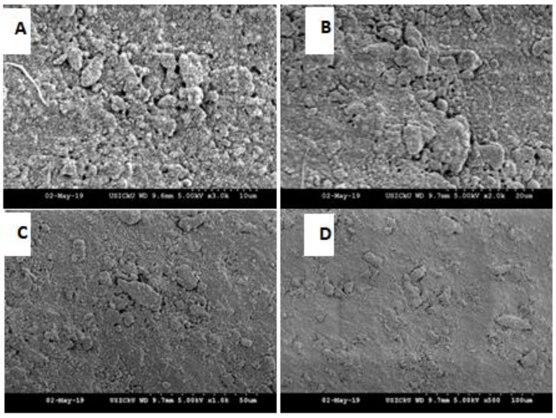

Figure 3: SEM image of surface of specimen after etching with 37% phosphoric acid concentrations after 10 minutes at (A)×500, (B) x1000, (C) x2000 and (D) x3000 magnification.

Group-IV

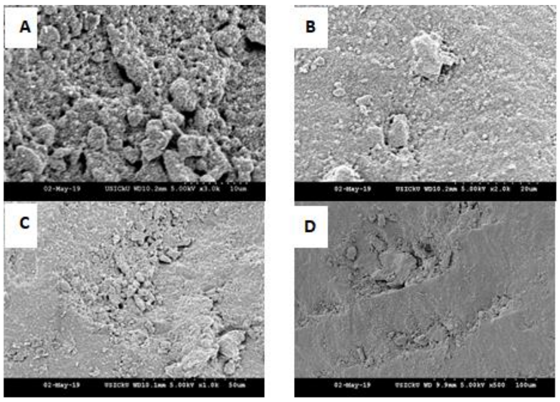

Figure 4: SEM image of surface of specimen after etching with 37% phosphoric acid concentrations after 15 minutes at (A)×500, (B) x1000, (C) x2000 and (D) x3000 magnification.

DISCUSSION

A lot of research has been focused on finding luting materials that offer adequate retention of a fixed dental prosthesis. However, not only are the physical characteristics of the luting cement responsible for the retentive phenomenon but also of considerable importance is the surface characteristics of the fitting surface of the prosthesis.[9] Prostheses fabricated from zirconia-ceramics are supposed to be more clinically durable than those prepared from other all-ceramic systems. A rough and irregular surface of yttria stabilized zirconia (3Y-TZP) creates micro-retentive depressions for the bonding systems, thus increasing the bonding surface as well as the wettability and surface energy.[24] Airborne-particle abrasion has been used in an attempt to enhance the surface area available for bonding: the treatment, however, appeared to be inadequate to establish reliable ceramic/ cement bonds.[25,26] In addition Kern et al and Yang et al also expressed concern that particle abrasion if carried out too aggressively could damage the surface causing a negative impact on the physical properties.[16,27] Alternative techniques like selective infiltration etching and use of hot chemical etching solutions have been proposed,[20] and have shown good results in achieving adequate surface roughness of the ceramic restoration.[21] Selective Infiltration Etching uses a heat-induced maturation process to pre-stress the surface grain boundaries in zirconia in order to allow infiltration of molten glass into these boundaries.[24] The glass is then etched out using HF, thereby creating a 3D network of inter-granular pores that allows Nano mechanical interlocking of resin cement.

Lee et al in their study demonstrated that higher concentrations and longer etching times using hydrofluoric acid (HFA) resulted in sufficient etching of the surface of zirconia restoration.[28] In a recent study, also, it has been seen that HF etching could induce the tetragonal-to-monoclinic phase transformation of zirconia due to low-temperature degradation.[29] Such effects are more prominent at higher temperatures and concentrations as pointed out by Guo et al and Xie et al.[30-32] According to Egilmez et al, the flexural strength of zirconia decreased significantly after immersion in 4% acetic acid at 80°C for 7 days.[33] In other studies, Y-TZP readily corroded after immersion in 100°C HCl/Fe2Cl3, hydrofluoric acid (HF), nitric acid, and sulfuric acid for 30 minutes.[21,34] These findings collectively suggest the potential adverse effects of acid solutions on the crystalline structure and mechanical properties of Y-TZP.

Thus the present study was aimed at testing an alternative protocol for etching zirconia based restorations. The use of 37% phosphoric acid has shown acceptable etching of the specimens as observed on electron micrographic examination. The extent of surface roughness is also directly dependent on the duration of exposure with longer etching times resulting in rougher surfaces. The quality of bonding after acid etching with phosphoric acid was, however, not tested clinically or under laboratory conditions. The effect on the chemical composition of the zirconia specimen was also not evaluated, nor were the physical properties before and after surface treatment evaluated.

Further research is needed to evaluate these factors. Acceptable etching with phosphoric acid, if obtained, will eliminate the need for use of more hazardous hydrofluoric acid, as has been reported in many studies about the toxic nature of fumes that result from HFA.[35] In addition to better handling properties, phosphoric acid provides the advantage of ease of availability, being available in almost every dental practitioner's office.

CONCLUSION

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Etching with a 37% Phosphoric acid solution can serve as an alternative method for roughening zirconia surfaces.
2. An increase in etching time has a positive effect on micro-morphology of the zirconia surface.
3. Long-term investigations are necessary to ensure the clinical efficacy of 37% phosphoric acid etching of zirconia for resin–zirconia bonding.

REFERENCES

1. Borchers L, Stiesch M, Bach FW, Buhl JC, Hubsch C, Kellner T, et al. Influence of hydrothermal and mechanical conditions on the strength of zirconia. Acta Biomater. 2010;6(12):4547-52.
2. Zhang Y, Lawn B. Fatigue sensitivity of Y-TZP to microscale sharp-contact flaws. J Biomed Mater Res Appl Biomater. 2005;72:388-92.
3. Papia E, Larsson C, du Toit M, et al. Bonding between oxide ceramics and adhesive cement systems: a systematic review. J Biomed Mater Res Part B Appl Biomater. 2014;102:395–413.
4. Ozcan M, Vallittu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. Dent Mater 2003;19:725-31.
5. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent 2003;89:268-74.
6. Lúty H, Loeffel O, Hammerle CHF. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. Dent Mater. 2006;22:195-200.
7. Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. Dent Mater. 1998;14:64-71.
8. Burke FJ, Fleming GJ, Nathanson D, Marquis PM. Are adhesive technologies needed to support ceramics? An assessment of the current evidence. J Adhes Dent. 2002;4:7-22.
9. Blatz MB, Chiche G, Holst S, Sadan A. Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia. Quintessence Int. 2007;38:745-53.
10. Ha, J.Y.; Son, J.S.; Kim, Y.K.; Kim, K.H.; Kwon, T.Y. Effect of heat treatment of dental zirconia ceramic treated with three different primers on the bonding durability of resin cement. Macromol. Res. 2013, 21, 71–77.
11. Aboushelib MN, Mirmohamadi H, Matinlinna JP, Kukk E, Ounsi HF, Salameh Z. Innovations in bonding to zirconia-based materials. Part II: Focusing on chemical interactions. Dent. Mater. 2009;25:989–993.

12. 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.
13. Kern M. Resin bonding to oxide ceramics for dental restorations. J. Adhes. Sci. Technol. 2009, 23, 1097–1111.
14. Jevnikar P, Krnel K, Kocjan A, Funduk N, Kosmač T. The effect of nano-structured alumina coating on resin-bond strength to zirconia ceramics. Dent. Mater. 2010, 26, 688–696.
15. Phark JH, Duarte SJ, Kahn H, Blatz M.B, Sadan A. Influence of contamination and cleaning on bond strength to modified zirconia. Dent. Mater. 2009, 25, 1541–1550.
16. Kern M, Barloi A, Yang B. Surface conditioning influences zirconia ceramic bonding. J. Dent. Res. 2009, 88, 817–822.
17. Wegner SM, Kern M. Long-term resin bond strength to zirconia ceramic. J. Adhes. Dent. 2000, 2, 139–147.
18. Noda M, Okuda Y, Tsuruki J, Minesaki Y, Takenouchi Y, Ban S. Surface damages of zirconia by Nd:YAG dental laser irradiation. Dent Mater J 2010; 29: 536-541.
19. Spohr AM, Borges GA, Junior LH, Mota EG, Oshima HM. Surface modification of In-Ceram Zirconia ceramic by Nd:YAG laser, Rocatec system, or aluminum oxide sandblasting and its bond strength to a resin cement. Photomed Laser Surg 2008; 26: 203-208.
20. 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; 98: 379-388.
21. Casucci A, Osorio E, Osorio R, Monticelli F, Toledano M, Mazzitelli C, Ferrari M. Influence of different surface treatments on surface zirconia frameworks. J Dent 2009; 37: 891-897.
22. Casucci A, Mazzitelli C, Monticelli F, Toledano M, Osorio R, Osorio E, Papacchini F, Ferrari M. Morphological analysis of three zirconium oxide ceramics: Effect of surface treatments. Dent Mater 2010; 26: 751-760.
23. Phark JH, Duarte S Jr, Blatz M, Sadan A. An in vitro evaluation of the long-term resin bond to a new densely sintered high-purity zirconium-oxide ceramic surface. J Prosthet Dent 2009; 101: 29-38.
24. 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
25. Guazzato M, Quach L, Albakry M, Swain MV. Influence of surface and heat treatments on the flexural strength of Y-TZP dental ceramic. J Dent 2005;33:9-18.
26. Sato H, Yamada K, Pezzotti G, Nawa M, Ban S. Mechanical properties of dental zirconia ceramics changed with sandblasting and heat treatment. Dent Mater 2008;27:408-14.
27. Yang B, Barloi A, Kern M. Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin. Dent. Mater. 2010, 26, 44–50.
28. Lee MH, Son JS, Kim KH, Kwon TY. Improved resin-zirconia bonding by room temperature hydrofluoric acid etching. Materials, 2015, 8, 850-866.
29. Sriamporn T. Thamronganankul, N.; Busabok, C.; Poolthong, S.; Uo, M.; Tagami, J. Dental zirconia can be etched by hydrofluoric acid. Dent. Mater. J. 2014, 33, 79–85.
30. Guo X. On the degradation of zirconia ceramics during low-temperature annealing in water or water vapor. J Phys Chem Solids. 1999; 60: 539–546.
31. Guo X, Schober T. Water incorporation in tetragonal zirconia. J Am Ceram Soc. 2004; 87: 746–748.
32. Xie H, Shen S, Qian M, Zhang F, Chen C, Tay FR (2015) Effects of Acid Treatment on Dental Zirconia: An In Vitro Study. PLoS ONE 10(8): e0136263. doi:10.1371/journal.pone.0136263
33. Egilmez F, Ergun G, Cekic-Nagas I, Vallittu PK, Lassila LV. Factors affecting the mechanical behaviour of Y-TZP. J Mech Behav Biomed Mater. 2014; 37: 78–87. doi: 10.1016/j.jmbbm.2014.05.013
34. Xie H, Chen C, Dai W, Chen G, Zhang F. In vitro short-term bond strength performance of zirconia treated with hot acid etching and primer conditioning. Dent Mater J. 2013; 32: 928–938.
35. Kirkpatrick JJ, Enion DS, Burd DA. Hydrofluoric acid burns: a review. Burns. 1995;21:483–493.

How to cite this article: Fayaz A, Bali SK, Bhat MM, Bashir A. Effect of 37% Phosphoric Acid Etchant on Surface Topography of Zirconia Crowns – An In Vitro Study. Ann. Int. Med. Den. Res. 2020; 6(5):DE04-DE07.

Source of Support: Nil, **Conflict of Interest:** None declared