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Bond strength of resin cements to zirconia ceramics: Influence of micromechanical roughening and chemical etching

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Abstract:
Purpose: The aim of this study was to evaluate the influence of combining sandblasting with chemical etching of zirconia surface on the shear bond strength at zirconia/resin cement interface.
Materials and methods: Ninety six zirconia discs were fabricated using CAD/CAM technology then divided according to aluminum oxide particles size used for sandblasting (50 and 110 µm) into two main groups (n=48) then each main group divided according to NH_4HF_2 etching protocol (170^{0}C for 10 min, 170^{0}C for 20 min and 190^{0}C for 10 min) to three subgroups (n=16). Zirconia discs were bonded to composite discs using resin cement (Multilink®N). 8 specimens of each treatment group were stored in water bath for one week and the other 8 specimens stored in water bath for 6 months followed by 10,000 thermal cycles then all specimens underwent shear bond strength testing.
Results: Two Way ANOVAs test showed no statistical significant difference between the two different aluminum oxide particles size used for sandblasting neither the different NH_4HF_2 etching protocols, after aging statistical significant difference appeared between different NH_4HF_2 etching protocols P (≤0. 05). (110 µm + 170^{0}C for 10 min) group had the highest mean value of bond strength before aging (34.0±11.4). Bond strength values of all test groups badly decreased after aging.
Conclusions: Combining sandblasting with chemical etching of zirconia surface provided sufficient bond strength to the resin cement that was badly affected after aging.
Clinical Significance: Etching zirconia surface by NH_4HF_2 after sandblasting increased surface roughness improving bond strength with resin cement.
Keywords: Zirconia, Surface treatment, Bond strength, Resin cement.

Introduction

Owing to the superior mechanical properties of yttria-stabilized tetragonal zirconia (Y-TZP) ceramics, it has become the predictable material for all ceramic restorations especially in areas with high functional forces.(1) Monolithic zirconia restorations without veneering ceramic was introduced to provide higher success rate of zirconia based restoration and solve the problem of porcelain veneer chipping.(2)

The cementation procedures strongly affects the clinical and long term success of the restoration,(3) cementation or luting of zirconia restorations was initially recommended using the conventional cements such as; zinc phosphate cements, glass ionomer cements or resin-modified glass-ionomer cements.(4) However, adhesive cementation using resin cements provides better retention, improved marginal adaptation preventing microleakage, high fracture resistance of both the restoration and the restored tooth with improved long term performance.(5)

Acid etching of the ceramic surface using hydrofluoric acid followed by silanation is the conventional method used for adhesive cementation which is not applicable for zirconia based ceramics as zirconia is a polycrystalline structure that lacks silica or glass phase essential for the etching by HF acid.(6)

The formation of micromechanical interlocking is essential to achieve strong and durable

bond between resin cement and zirconia,(7) different surface treatments have been developed to achieve this mechanical interlocking including**;** airborne particle abrasion, laser application, slelective infiltration etching, acid etching of zirconia surface or combination of these methods. Reaching stable and reliable resin cement bond to Y-TZP zirconia still a challenge.(8)

Concerning zirconia surface treatment, airborne particle abrasion or sandblasting is considered a key factor for gaining micromechanical retention with resin cement.(9) Sandblasting of zirconia surface with Al_2O_3 particles cleans the surface by removing any organic contaminations, provides surface micro roughness and improves surface wettability.(10)

Different sizes between 20 and 250 µm of the aluminum oxide particles have been used for zirconia sandblasting

with 50 and 110 μm being the most commonly used.[11] The rough surface
produced by sandblasting of zirconia surface allows the penetrations of resin cement into the micro-retentions on the surface providing a strong mechanical interlock.[12]

Many studies reported that sandblasting enhanced the bond strength between Y-TZP ceramics and resin cement compared with untreated non-sandblasted groups.[13,14] However, some studies showed that the surface roughness produced by sandblasting may be not sufficient enough to improve bond strength with the resin cement,[15,16] combination of sandblasting with other methods of zirconia surface treatment including acid etching have been introduced.

Acid etching of zirconia with ammonium hydrogen difluoride was recently introduced and it has been demonstrated that it had the ability of etching zirconia surface providing a rough surface and exposing zirconia grains which would allow mechanical interlocking with the resin cement.[17,18] The purpose was of this study was to evaluate the effect of combining sandblasting and acid etching of zirconia surface on shear bond strength between resin cement and zirconia ceramics with and without artificial aging.

MATERIALS AND METHODS:

A total number of 96 Y-TZP ceramic discs were fabricated using CAD/CAM technology to the dimensions of 8 mm diameter and 3 mm thickness; the zirconia discs were milled from zirconia blank (Ceramill® Zolid 71 XS Blank) then sintered in high temperature furnace according to manufacturer's recommendations. 96 composite resin discs were fabricated using (Nexcomp, META® BIOMED, Korea) in the dimensions of 4 mm diameter and 3 mm thickness. The 96 zirconia discs were divided according to aluminum oxide particles size used for sandblasting (50

and 110 μm) into two main groups (n=48) then each main group divided according to different

etching protocols by NH4HF2 (170^{0}C for 10 min, 170^{0}C for 20 min and 190^{0}C for 10 min) to

three subgroups (n=16). Etched specimens were rinsed in water, ultrasonically cleaned in ethanol (ethyl alcohol 95%) for 15 min and air dried before bonding. The composite resin discs were bonded to previously treated zirconia discs using multistep adhesive resin cement (Multilink®N, Ivoclar Vivadent, Liechtenstein) according to the manufacturer instructions without application of any priming agent. After specimens bonding, 8 specimens of each treatment group kept in water bath at 37^{0}C for one week and the other 8 specimens kept in water bath at 37^{0}C for 6 months followed by 10,000 thermal cycles using thermocycling device (Julabo®FT200, Germany). Following artificial aging procedures all the bonded specimens were fixed in self-cured acrylic resin surrounded by thermoplastic rings using a specially designed Teflon mold prior to shear bond strength testing. The shear bond strength testing of all bonded specimens was done using universal testing machine (Instron Industrial products, Norwood, USA). Failure patterns of debonded specimens were examined using binocular optical microscope and representative specimens for each failure pattern were further examined using SEM (Scanning Electron Microscope) (Quanta 250-FEG, FEI, Netherlands). For studying the surface topography of surface treated zirconia specimens, one specimen from each group was examined using SEM. Figure (1)

RESULTS:

Two-way ANOVA test showed no statistical significant difference between the two different aluminum oxide particles size used for sandblasting neither the different NH4HF2 etching protocols, after aging statistical significant difference appeared between different NH4HF2 etching protocols [P=.004]. One-way ANOVA test was used for shear bond strength (MPa) comparison after different surface treatments; mean SBS values were higher in all groups of short term storage than long term storage. In the long term storage, the highest mean SBS was recorded in samples etched at 170^{0}C for 10 minutes after sandblasting using both sizes of aluminum oxide particles (50 and 110 μm)[4.5±2.9 and 4.4±3.5] . The same results were found in short term storage with 110 micron group [34.0±11.4] slightly higher than 50 μm group [31.3±12.3] followed by samples etched at 190^{0}C for 10 min with 110 aluminum oxide particles

[30.2±13.1] Table (1). Post Hoc Tukey test that was used for Pairwise comparison between different test groups following Two-way ANOVA test revealed that there was no significant differences found between the different test groups.

DISCUSSION:

Damage to a zirconia frame was rarely reported with ceramic veneer chipping being the most frequent failure reason of zirconia-based restorations,(19) monolithic zirconia restorations solved the problem of porcelain veneer chipping with improved clinical and laboratory results demonstrated in many studies.(2,19) In the present study monolithic zirconia system (Ceramill® Zolid) was used to fabricate the zirconia specimens using CAD/CAM technology.

In the present study the zirconia specimens were bonded to composite discs instead of dental tissues. The homogeneous structure of the composite discs would prevent interpreting errors in the bond strength data that could happen with using dental tissue due to the heterogeneous microstructure of dentin.(20)
Shear stresses are the main factors affecting adhesion in clinical situations that can cause failure to the bonding of the restorative materials.(21) In the present study shear bond strength test (SBS) is used evaluate the effectiveness of resin cement bonding to zirconia ceramics, it has the advantages of being fast and easy and is the most commonly used bond strength test.(22)

There is still no consensus regarding the optimal size of Al2O3 particle that can be used for sandblasting of zirconia ceramics surface to achieve the maximum bond strength with resin cement.(23) Moon et al.(24) recommended sandblasting with 50 µm aluminum oxide particles as 50 µm they produced sufficient surface roughness without affecting the flexural strength of zirconia. Su et al.(25) recommended sandblasting using aluminum oxide particles size of 110 µm to obtain the highest bond strength to resin cement, 110 µm aluminum oxide particles produced a rougher surface of zirconia than 50 µm particles achieving higher bonding strengths. Chintapalli et al.(26) recommended sandblasting using aluminum oxide particles size less or equal to 110 µm to maintain the flexural strength of zirconia based restorations and to obtain strong bond strength with resin cement.

Reaching the optimal protocol for sandblasting of zirconia surface to obtain the maximum bond strength with resin cement remains a challenge.(12) In the present study we compared the effect of sandblasting with two different aluminum oxide particles size (50 and 110 µm) in combination with chemical etching on the bond strength between zirconia specimens and resin cement.
According to some studies sandblasting produced only slightly rough surface that was not sufficient enough to improve bond strength between zirconia ceramics and resin cement,(15,16) the present study tried to evaluate the effect of combining sandblasting treatment with other method of zirconia surface treatment (etching with ammonium hydrogen difluoride) on the bond strength of resin cement to zirconia ceramics.

Ruyter et al.(17) evaluated the effect of etching zirconia surface with NH4HF2 and found that it created a rough surface exposing zirconia grains with deep grooves, etching of zirconia surface with NH4HF2 produced higher bond strength with resin cement than sandblasting only. These results are in agreement with the results of another study by Akazawa et al.(18)

In Ruyter et al.(17) study NH4HF2 was applied as powder and aqueous slurries with no statistically significant difference, where in Akazawa et al.(18) study NH4HF2 was applied as powder. In the present study NH4HF2 was applied as aqueous slurries that can be applied on curved surfaces.

Ammonium hydrogen difluoride has a low melting point of 125^{0}C, in both Ruyter et al.(17) and Akazawa et al. (18) studies heating of the zirconia specimens after NH4HF2 application was

done at 170^{0}C for 10 minutes. In the present study we tried to compare the effect of increasing

both temperature and duration of heating on the bond strength of zirconia specimens to the resin

cement with three different etching protocols; heating at 170 ^{0}C for 10 minutes, heating at 170^{0}C for 20 minutes and heating for 190^{0}C for 10 minutes.
Artificial aging can stimulate intraoral conditions and is important to determine the durability of the bond obtained between resin cements and Y-TZP ceramics.(27,28) In the present study long-term water storage 37°C for six months and thermal cycling done for 10,000 cycles were used to stimulate aging and to compare bonding strengths before and after the aging process.

Regarding shear bond strength results a range between 10 and 13 MPa was considered the minimum clinically acceptable bond strength;(29,30) the combination of sandblasting with etching of zirconia surface by ammonium hydrogen difluoride produced a sufficient bond strength between the zirconia specimens after short term storage (one week in water bath) with a minimum bond strength value of (24.4±5.88) in (50 µm + etching at 170^{0}C for 20 min) Group

and maximum bond strength value of (34.0±11.4) in (110 µm + etching at 170^{0}C for 10 min) Group.

These results are in agreement with the results of Ruyter et al.(17) study where etching of zirconia surface with NH4HF2 slurries without silane treatment application also produced a sufficient bonding strength (37.3±11.5) however, in Ruyter et al.(17) study no prior sandblasting treatment was done before etching by NH4HF2 and shear bond strength testing was done only after 24 hours of bonding with no water storage.

In short term storage there was no statistical significant difference between the two different aluminum oxide particles size used for the sandblasting treatment neither the different NH4HF2 etching protocols used for etching of zirconia surface. However, after aging (long term water storage + thermocycling) a statistical significant difference appeared between different NH4HF2 etching protocols.

Regarding SEM imaging, the obtained images supported our results. Etching by ammonium hydrogen difluoride changed surface topography to rough surfaces with homogeneous grains and random porosities with different etching protocols produced similar images of the homogeneous grains, however similar SEM images were obtained by Ruyter et al.(17) and Akazawa et al.(18) where etching of zirconia surface by NH4HF2 was done without prior sandblasting treatment.

Regarding artificial aging, shear bond strength values decreased badly after long term water

storage and thermocycling treatment with bond strength value as low as (1.6±1.3) in (110 µm + etching at 170^{0}C for 20 min) Group and a highest bond strength value of (4.5±2.9) in (50 µm + etching at 170^{0}C for 10 min) Group.
Artificial aging decreased the bond strength between zirconia specimens and the resin cement; these results are in agreement with results of several other studies.(27,28,30)
Regarding failure pattern analysis of debonded specimens, failure patterns before aging were mainly mixed with some cohesive failure pattern. However, after artificial aging the

failure patterns were mainly adhesive without any cohesive failure patterns. Failure pattern analysis is important to assess the bond strength, cohesive and mixed failure patterns indicate high bond strength on the other hand adhesive failure patterns are usually associated with insufficient bond strength.(31)

CONCLUSION:

The following conclusion was found under the conditions of this study; combining sandblasting with etching of zirconia surface using ammonium hydrogen difluoride produced a sufficient bond between Y-TZP ceramics and resin cement, however the bonding strength was badly affected by artificial aging.

References

1. *Agustín-Panadero R, Román-Rodríguez JL, Ferreiroa A, Solá-Ruíz MF, Fons-Font. Zirconia in fixed prosthesis. A literature review. J Clin Exp Dent. 2014 Feb 1; 6(1):e66-73.*

2. *Özkurt-Kayahan Z. Monolithic zirconia: A review of the literature. Biomedical Research. 2016; 27 (4): 1427-1436.*

3. *Donovan TE. Factors essential for successful all-ceramic restorations. J Am Dent*

4. *Murthy V, Manoharan, Balaji, Livingstone D. Effect of four surface treatment methods on the shear bond strength of resin cement to zirconia ceramics- a comparative in vitro study. J Clin Diagn Res. 2014 Sep; 8(9):ZC65-8.*

5. *Usumez A, Hamdemirci N, Koroglu BY, Simsek I, Parlar O, Sari T. Bond strength of resin cement to zirconia ceramic with different surface treatments. Lasers Med Sci. 2013 Jan; 28(1):259-66.*

6. *Daou EE. The zirconia ceramic: strengths and weaknesses. Open Dent J. 2014 Apr 18;*

8:33-42.

7. *Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/cementation to zirconia and other non-silicate ceramics: where are we now? Dent Mater. 2011 Jan; 27(1):71-82.*

8. *Melo RM, Souza RO, Dursun E, Monteiro EB, Valandro LF, Bottino MA. Surface Treatments of Zirconia to Enhance Bonding Durability. Oper Dent.2015 Nov-Dec; 40(6):636-43.*

9. *Yi YA, Ahn JS, Park YJ, Jun SH, Lee IB, Cho BH, Son HH, Seo DG.The effect*

of sandblasting and different primers on shear bond strength between yttria-

tetragonal zirconia polycrystal ceramic and a self-adhesive resin cement. Oper Dent. 2015 Jan-Feb; 40(1):63-71.

10. *Mahmoodi N, Hooshmand T, Heidari S, Khoshro K. Effect of sandblasting, silica coating, and laser treatment on the microtensile bond strength of a dental zirconia ceramic to resin cements. Lasers Med Sci. 2016 Feb; 31(2):205-11.*

11. *Erdem A, Akar GC, Erdem A, Kose T. Effects of different surface treatments on bond strength between resin cements and zirconiaceramics. Oper Dent. 2014 May-Jun; 39(3):E118-27.*

12. *Aurélio IL, Marchionatti AM, Montagner AF, May LG, Soares FZ. Does air particle abrasion affect the flexural strength and phase transformation of Y-TZP? A systematic review and meta-analysis. Dent Mater. 2016 Jun; 32(6):827-45.*

13. *He M, Zhang Z, Zheng D, Ding N, Liu Y. Effect of sandblastingon surface roughness of zirconia-based ceramics and shear bond strength of veneering porcelain.*

Dent Mater J. 2014; 33(6):778-85.

14. *Gomes AL, Castillo-Oyagüe R, Lynch CD, Montero J, Albaladejo A. Influence of sandblasting granulometry and resin cement composition on microtensile bond strength to zirconia ceramic for dental prosthetic frameworks. J Dent. 2013 Jan; 41(1):31-41.*

15. *Oyagüe RC, Monticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Influence of surface treatments and resin cement selection on bonding to densely-sintered zirconium-oxide ceramic. Dent Mater. 2009 Feb; 25(2):172-9.*

16. *Kern M. Resin bonding to oxide ceramics for dental restorations. J Adhes Sci Technol. 2009; 23:1097-111.*

17. *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.*

18. *Akazawa N, Koizumi H, Nogawa H, Kodaira A, Burrow MF, Matsumura H. Effect of etching with potassium hydrogen difluoride and ammonium hydrogen difluoride on bonding of a tri-n-butylborane initiated resin to zirconia. Dent Mater J. 2019 Jul 31; 38(4):540-546.*

19. *Kontonasaki E, Rigos AE, Ilia C, Istantsos T. Monolithic Zirconia: An Update to Current Knowledge. Optical Properties, Wear, and Clinical Performance. Dent J (Basel). 2019 Sep 2; 7(3):90.*

20. Keshvad A, Hakimaneh SMR.Microtensile Bond Strength of a Resin Cement to Silica-Based and Y-TZP Ceramics Using Different Surface Treatments. J Prosthodont. 2018 Jan; 27(1):67-74.

21. El Mourad AM. Assessment of Bonding Effectiveness of Adhesive Materials to Tooth Structure using Bond Strength Test Methods: A Review of Literature. Open Dent J. 2018 Sep 28; 12:664-678.

22. Sirisha K, Rambabu T, Shankar YR, Ravikumar P. Validity of bond strength tests: A critical review: Part I. J Conserv Dent. 2014 Jul; 17(4):305-11.

23. Garcia Fonseca R, de Oliveira Abi-Rached F, dos Santos Nunes Reis JM, Rambaldi

E, Baldissara P. Effect of particle size on the flexural strength and phase transformation of an airborne-particle abraded yttria-stabilized tetragonal zirconia polycrystal ceramic. J Prosthet Dent. 2013 Dec; 110(6):510-4.

24. Moon JE, Kim SH, Lee JB, Han JS, Yeo IS, Ha SR. Effects of airborne-particle abrasion protocol choice on the surface characteristics of monolithic zirconia materials and the shear bond strength of resin cement. Ceramics International. Jan 2016; 42(1): 1552-1562.

25. Su N, Yue L, Liao Y, Liu W, Zhang H, Li X, Wang H, Shen J. The effect of various sandblasting conditions on surface changes of dental zirconia and shear bond strength between zirconia core and indirect composite resin. J Adv Prosthodont. 2015 Jun; 7(3):214-23.

26. Chintapalli RK, Mestra Rodriguez A, Garcia Marro F, Anglada M. Effect of sandblasting and residual stress on strength of zirconia for restorative dentistry applications. J Mech Behav Biomed Mater. 2014 Jan; 29:126-37.

27. Noda Y, Nakajima M, Takahashi M, Mamanee T, Hosaka K, Takagaki T, Ikeda

M, Foxton RM, Tagami J. The effect of five kinds of surface treatment agents on the bond strength to various ceramics with thermocycle aging. Dent Mater J. 2017 Nov 29; 36(6):755-761.

28. Gomes AL, Ramos JC, Santos-del Riego S, Montero J, Albaladejo A. Thermocycling effect on microshear bond strength to zirconia ceramic using Er:YAG

and tribochemical silica coating as surface conditioning. Lasers Med Sci.2015 Feb;30(2):787-95.

29. Capa N, Ozkurt Z, Canpolat C, Kazazoglu E. Shear bond strength of luting agents to fixed prosthodontic restorative core materials. Aust Dent J. 2009 Dec; 54(4):334-40.

30. Attia A. Bond strength of three luting agents to zirconia ceramic - influence of surface treatment and thermocycling. J Appl Oral Sci. 2011 Aug; 19(4):388-95.

31. Hipólito VD, Alonso RC, Carrilho MR, Anauate Netto C, Sinhoreti MA, Goes MF. Microtensile bond strength test and failure analysis to assess bonding characteristics of different adhesion approaches to ground versus unground enamel. Braz Dent J. 2011; 22(2):122-8.

Table (1): Effect of temperature/time change on shear bond strength at maximum load.

		Shear stress at maximum load (Mpa) Temperature /time			One Way ANOVA test
		170 ° for 10 min	**170 ° for 20 min**	**190 ° for 10 min**	
Sandblasting 50 micron	**Long term storage**	4.5±2.9	1.8±2.23	2.2±2.0	F=2.96 P=0.07
	Short term storage	31.3±12.3	24.4±5.88	25.1±5.7	F=1.59 P=0.23
Sandblasting 110 micron	**Long term storage**	4.4±3.5	1.6±1.3	2.0±1.7	F=3.26 P=0.06
	Short term storage	34.0±11.4	26.6±8.6	30.2±13.1	F=0.88 P=0.43

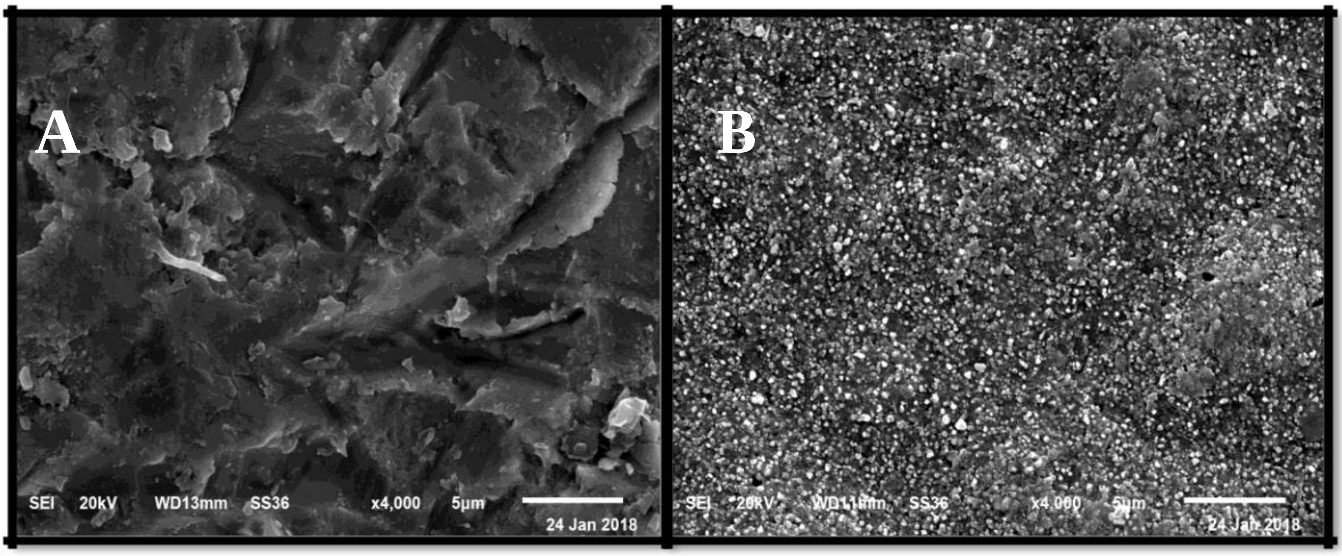

Figure (1): A) SEM imaging at ×4000 magnification of zirconia surface after sandblasting treatment.

B) SEM imaging at ×4000 magnification of zirconia surface after etching by NH_4HF_2