IOSR Journal of Dental and Medical Sciences (IOSR-JDMS)
e-ISSN: 2279-0853, p-ISSN: 2279-0861.Volume 18, Issue 3 Ser. 15 (March. 2019), PP 80-85
www.iosrjournals.org

Microtensile Bond Strength of Zirconia Ceramics with Different Surface Treatments

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Abstract
Objectives: *The aim of this in-vitro study was to evaluate the hydrolytic stability of different surface treatments on the bond strength of yttrium-oxide-partially-stabilized zirconia ceramics.*
Materials and Methods: *InCoris TZI C (Sirona Dental Systems GmbH, Germany) zirconia blocks were sintered according to the manufacturer's instructions and randomly divided into three groups according to the surface treatment methods as follow;Group I: Air particle abrasion, Group II: Air particle abrasion followed by piranha acid etch solution, and Group III: Air particle abrasion followed by hot acid etching solution. Zirconia blocks were bonded to the pre-constructed composite blocks using Panavia SA cement Plus. Microbars from each subgroups were randomly selected, half of them were subjected to the testing procedures without thermalcycling, while the other half were subjected to 10,000 thermalcycles. Microtensile bond strength testwas performed using a universal testing machine (0.5 mm/min).*
Results:*There was, a significant difference (P<0.05) between the three main surface treatment.*
Conclusion: *The bond strength was influenced by the different surface treatment methods, and aging process.*
Key words: *Dental ceramics;Surface treatment; Resin cement; Bond strength.*

Date of Submission: 12-03-2019 Date of acceptance: 28-03-2019

I. Introduction

The technological evolution of dental ceramics restorations has been distinguished over the last decades. From feldspathic porcelains to zirconia-based all-ceramics, fabulous progress has been made in terms of mechanical performance, with increase in fracture toughness and flexural strength.[1]) Zirconia ceramics are glass-free polycrystalline microstructure, non etchable, low surface energy, and chemically inert with few hydroxyl groups for covalent bonding.[2,3]Therefore, the main limitation with zirconia ceramics is their limited ability to bond with resin cements which reduces the clinical durability.(4)There are several methods for achieving a high bond strength with Y-TZP, but this bond strength should withstand in the surrounding oral environment over years.[5]The combined mechanical and chemical surface pretreatment of zirconia improved the bond durability between resin cements and zirconia.[6] Surface cleaning, surface roughening, and surface wettability are essential before the application of resin cements for achieving a strong durable bond strength.[7]

Various surface treatment methods are used nowadays such as tribochemical silica coating, sandblasting, grinding, glass micro pearls coating, acid etching, plasma spraying, glaze layer coating, coating with nano structured alumina, silane application, selective etching techniques, and laser etching have been evaluated on zirconia restoration.[8]

Airborne-particle abrasion is one of the most common surface treatment method. This method increases the surface energy, wettability, surface roughness, and provides micromechanical undercuts therefore improves the adhesion of zirconia ceramic.[9]The effects of this technique on the mechanical properties of zirconia are argumentative. Some demonstrated that the flexural strength increase as the sandblasting method induce t⇒m transformation and arrest crack propagation.[10,11] However, other reported that sandblasting results in structural defects and induction of sharp cracks that enhance radial cracking during function.[12]Piranha solution is known as a cleaning reagent, and composed of hydrogen peroxide and sulfuric acid (H_2O_2:H_2SO_4).[2] This solution is a strong oxidizing corrosive agent used to remove the organic impurities and hydroxylate surfaces, resulting in improving the surface hydrophilicity.[13]Hot acid etching is another surface treatment which has the advantage of less generated internal stress and using lower temperature compared with other surface roughening methods, such as laser etching, applied glaze layer, sandblasting, and selective infiltration etching. The hot etching solution improves the surface roughness through a corrosion controlled process. This

technique is based on removing the less well arranged and high energy peripheral atoms resulting in wider grain boundaries.[14]

Resin cements were introduced as an alternative to the acid base reaction cements and based on dimehacrylate resin chemistry.[15]Based on bonding mechanism resin cements can be classified into two main groups conventional resin cements and self adhesive resin cements.[16,17] The self adhesive resin cements adhere to the tooth structure without any surface preconditioning.[18,19]This type of cements contain acidic functional monomers that demineralize the tooth structure and bind with calcium in the hydroxylapatite to create a strong bonding between the tooth and the methacrylate network.[20]It has been concluded that the MDP-containing resin cements have the highest bond strength with the abraded zirconia. This is due to the chemical interaction between the functional phosphoric acid monomer and the hydroxyl groups of the passive zirconia surface.[21,22]

There are various testing methods to study the bonding strength of resin based materials to zirconia ceramics, such as macroshear, microshear, macrotensile and microtensile tests. Considering the microtensile test, the small interfacial bonding zone ($1mm^2$) shows more homogeneous stress distribution, therefore more sensitive evaluation of bond performances when specimens are aligned correctly.[23,24]

Thus, the objective of this study was to evaluate the effect of different surface treatments and MDP-containing resin cement on the bond strength of yttrium-oxide-partially-stabilized zirconia ceramic. The research hypotheses were a) the different surface treatments would not improve bond strength; b) thermocycling would decrease the bond strength.

II. Material And Methods

Materials used in this study are listed in Table (1). The partially sintered zirconia block InCoris TZI C(Sirona Dental Systems GmbH, Germany) was cut into blocks (10 mm length × 10 mm width × 6 mm thickness) using a precision cutting machine (Isomet 4000, Buehler Ltd, Lake Bluff, IL, USA), and were sintered according to the manufacturer recommendations. The surface to be bonded was polished forall specimens using 1000-grit silicon abrasive paper, ultrasonically cleaned with (Baku 3550, China) for 15 min in distilled water, and air dried.

The light cured composite resin Tetric N-Ceram(IvoclarVivadent, Liechtenstein, Germany) was incrementally condensed in a specially designed Teflon split moldwith the same dimensions of zirconia blocks. Each layer of the composite resin was light cured according the manufacturer's instructions for 20seconds using a LED curing unit (Gulin Woodpecker Medical Instrument Co, Ltd, Guangxi, China). The surface to be bonded was polished forall specimensusing 400- grit silicon abrasive paper then ultrasonically cleaned, and air dried.

Table (1): Materials used in this study.

Product name	Composition	Patch number	Manufacturer
InCoris TZI C	(ZrO_2) 87-89%, (Y_2O_3) 4.5- 6%, (HfO_2) ≤ 5%, (Al_2O_3) ≤ 0.04%, Other oxides ≤ 1.1	2017082018	Sirona Dental Systems GmbH, Germany
Tetric N-Ceram	Dimethacrylates, barium glass, ytterbium trifluoride, mixed oxide	W84583	IvoclarVivadent, Liechtenstein, Germany
Panavia SA Cement Plus Automix	• Paste A: 10MD, HEMA, Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate • Paste B:Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate	8N0111	Kuraray Noritake Dental, Tokyo, Japan
Aluminum oxide particle	50 µm AL_2O_3		Cobra, Renfert GmbH, Hilzingen, Germany
Hot etching solution	69% nitric acid, 48% hydrofluoric acid		Non- commercial
Piranha etching solution	96% sulfuric acid, 30% hydrogen peroxide		Non- commercial

Surface treatments of the ceramic blocks

Zirconia specimens were randomly divided into 3 main groups according to the surface treatment that were established as follow;

Group I: Airborne-particle abrasion;The bonding surface was air particle abraded with 50 µm aluminum oxide particles (Al_2O_3 Cobra, Renfert GmbH, Hilzingen, Germany) at a pressure of 0.2 MPa for $10sec/cm^2$ at a distance of 10 mm perpendicular to the ceramic surface.

Group II: Airborne-particle abrasion+ piranha solution;The bonding surface was air particle abraded with Al_2O_3 as described for Group I. The bonding surface was then immersed in piranha acid etching solution a mixture of 96% sulfuric acid (H_2SO_4) (Al Nasr pharmaceutical chemicals Co., ADWIC, Egypt) and 30% hydrogen peroxide (H_2O_2) from (Piochem Co., Egypt) with a ratio of (3: 1), respectively for four days.

Group III: Airborne-particle abrasion+ hot etching solution;The bonding surface was air particle abraded with Al_2O_3 as described for Group I. The bonding surface was then immersed in hot chemical etching solution a mixture of 69% nitric acid (HNO_3) from (Honeywell International Inc., Burdick and Jackson, Seelze, Germany) and 48% hydrofluoric acid (HF) from (Honeywell International Inc., Riedel-de Haën, Seelze, Germany) with a ratio of ($1HNO_3$: 1HF). The solution was heated up to 100°C in a water bath for 25 min. All treated zirconia blocks were then rinsed with distilled water, ultrasonically cleaned with in a distilled water for 10 min, and air dried.

Bonding of the composite blocks to zirconia specimens

The bonding procedures were performed immediately after each surface treatment to eliminate any possible surface contamination. The composite blocks were bonded to the bonding surfaces of zirconia ceramic blocks using MDP-containing self adhesive resin cement (Panavia Sa,Kuraray Noritake Dental, Tokyo, Japan)under a static load of 1kg for 5 min.The resin cement was light cured from all directionsaccording the manufacturer's instructions using a LED curing unit for10 sec (Gulin Woodpecker Medical Instrument Co, Ltd, Guangxi, China).

Preparation of specimens for testing

The ceramic/ resin/ composite assemblies were then stored in a distilled water at 37°C for 24 hours before cutting procedures. The assemblies of each group were sectioned perpendicular to the bonding interface area to obtain slabs of $1mm^2$ thickness using a diamond blade (Buehler, wafering blade, 20LC, 11e4225, USA). The assemblies were rotated 90° and bonded again to the metallic base, the cutting process was repeated again perpendicular to the bonding interface area, and microbars with $1mm^2$ thickness were obtained. These microbars were examined under a stereomicroscope (MA 100 Nikon stereomicroscope, Japan) at 50x magnification for selecting the intact specimens that were free from any microcracks.

Twenty microbars from each groups were randomly selected, half of these microbars were subjected to the testing procedures without thermalcycling, while the other were subjected to 10,000 thermal cycles. This process was achieved at a temperature ranging between 5°C and 55°C, with a dwelling time of 30 sec in each water bath and transferring time of 5 sec

Microtensile bond strength test

A specially designedattachment was fabricated to permit a precise alignment of the microbar with the applied force during testing. Each microbar was glued inplace from the outer edges using one drop of a cyanoacrylate glue (AmirA2000, oc-cyanoacrylate adhesive, china), and the bonding interface must be free from glue. The attachment was then mounted into the universal testing machine (3345, Instron, 2519e104, 3345, Canton, MA, USA), and a tensile force was applied at a crosshead speed of 0.5 mm/min until debonding of the specimen occurred. A mean value of microtensile bond strength for each specimen was calculated in MegaPascal (Bluehill Lite software, Instron, MA, USA) by dividing the load at failure (N) by the adhesive area (mm^2).

Failure and statistical analysis

The fractured specimens were observed under a steriomicroscope (MA 100 Nikon steriomicroscope, Japan) at 50x magnification to determine the mode of failure. The mode of failure was recorded as follow: adhesive failure in the interface between resin cement and ceramic, cohesive failure within composite, cohesive failure within ceramic, and mixed failure combination of adhesive and cohesive failure mode.Data were tabulated, coded then analyzed using IBM SPSS (Statistical Package for social Science) computer software version 22.

III. Results

Mean±standard deviation values of μTBS without and after aging are summarized in (Table 2). The results of two-way ANOVA- Omnibus test of variance exhibited that the main surface treatmentand the aging significantly affect the bond strength ($P< 0.05$). Without aging; Group I showed the highest bond strength (33.491± 2.76) followed by, Group III(27.486± 6.42), and then group II(22.072± 3.75). Group III recorded the highest bond strength(25.120±4.55) after aging.The mode of failure of all tested groups without aging and after aging was demonstrated in (Table 3). The mode of failure was predominantly cohesive within ceramic or

composite for the tested groups without aging, while after aging was predominantly adhesive between the resin cement and ceramic with less cohesive failure.

Table (2): Means and standard deviations (±SD) of the μTBS for the tested groups without and after aging.

Tested groups	Without aging	After aging
Group I	33.491± 2.76	21.516±7.33
Group II	22.072± 3.75	23.077±5.11
Group III	27.486± 6.42	25.120±4.55

Table (3**):** Failure mode of all tested groups without aging and after aging.

Tested groups	Without aging			After aging		
	A	**C**	**M**	**A**	**C**	**M**
Group I	2	6	2	6	2	2
Group II	4	4	2	4	2	4
Group III	2	6	2	4	2	4

A: Adhesive failure in the interface between resin cement and ceramic.
C: Cohesive failure within composite or cohesive failure within ceramic.
M: Mixed failure combination of adhesive and cohesive failure mode.

IV. Discussion

This study evaluated the effect of several combined surface treatment methods on the strength of bonding between zirconia and resin cement. The results of this study reject the null hypothesis, because various surface treatmentsaffected the μTBS.

The microtensile bond strength test is more reliable than shear and tensile bond strength tests.[25]Shear bond strength is a popular test due to it is easy applied procedures. However this test often results in cohesive fractures because of in homogenous stress distribution at the bonding interference area.[26]Regarding the traditional tensile strength the difficulty of the specimens alignment also may results in uneven stress distribution. Microtensile test was applied here with a specially designed attachment for parallel alignment of specimens. This resulting in more uniform stress distribution during loading that leads to more reasonable estimation of bond strength and fewer cohesive failures.[27,28]

The results of this study exhibited that there was a significant difference between the three surface treatments. Group I showed the highest bond strength without aging followed by Group III and then Group II.These results were agree with Moradabadi et al.,[29]who observed that the specimens treated with airborne particle abrasion recorded the higher bond strength than the group treated with APA and (HF/HNO_3) etching solution at room temperature for 2 min. Also revealed that airborne particle abrasion under SEM produced jagged and wide distribution roughness on the zirconia surface resulting in micromechanical retention and so better bonding strength. However, by adding the etching solution to the abraded zirconia surface leading to deformation of this jagged roughness to be rounded and had hump surface topography. This deformation is due to the dissolution of higher surface energy grain boundaries so the reduction in micromechanical retention. Another studies also showed that the using of MDP-containing resin cement improved the bond strength with APA.[26,30]Also these results were in agreement with Zandparsa et al.,[31] who found that the bond strength of piranha acid etching solution was significantly lower than the APA. There explanation is that this etching solution cleans and hydroxylates the surface without undercuts formation which are particularly important in micromechanical interlocking formation with the cement. Another study showed higher bond strength with APA than piranha solution[13].In contrary**,**Lahbour et al.,[2] reported that the addition of piranha acid solution to the abraded zirconia had the higher bond strength than APA using 110μm particle size. The possible explanation is that the aggressive air abrasion using bigger grain particles size could result in ditching between resin cement and zirconia surface.[32]

Group I showed significant decrease in bond strength after aging, while Group III showed the highest bond strength after aging. The possible explanation is that hot acid etching improved the surface roughness by dissolving the less well arranged peripheral atoms of zirconia surface resulting in larger grain boundaries formation which increase the mechanical interlocking with resin cement with no phase transformation.[27,33] Moreover, the hydrolytic stability of the functional monomer (10 MDP) due to the presence of a long carbonyl chain.[34]The significant decrease of bond strength Group I after aging may be related to the air-borne particle abrasion resulted in structural defects such as voids and flaws that enhance radial cracking during function.[12]These results in agreement with Xie et al.,[35] who observed that the hot acid etched group showed the highest bond strength after aging compared to the APA group. Also they revealed that under SEM the hot acid etching improved the surface roughness, and removed the superficial ceramic layer resulting in a homogeneous granular and porous texture. Another study also showed higher bond strength with hot etching than APA after aging [(27)] These results were partially in agreement with Mahmoodi et al.,[36]who observed that

the APA specimens bonded with self adhesive resin cement(Clearfil SA) exhibited debonding prior testing. This may be related to that the Zirconia primer wasn't applied to zirconia surface prior to bonding procedures according to the manufacture's recommendations. This primer contains methacrylate group that copolymerized with the resin cement, and the Phosphoric acid group bonded to zirconia surface.

The results of this present study revealed that the bonding strength between resin cements and zirconia ceramic reduced after aging. These results were in agreement with others.[13,25,27,36] The possible explanations for this could be related to many factors such as, The microbars used in this study exposed a wide surface area of the bonding interface to the effect of thermocycling. However with shear bond strength test only the boundaries of the bonding interface exposed to this effect.[5,37]Also the expansion of the cement layer due to water absorption results in degradation of the resin cement.[25,35] Furthermore the mismatching between the coefficient of thermal expansion of the composite resin, resin cement, and zirconia ceramics of the bonded complex.[19,25]

The mode of failure of the specimens tested without aging was predominantly cohesive with in ceramic or composite, while after aging was predominantly adhesive between the resin cement and ceramic, and some specimens showed mixed failure with less cohesive failure. In this present study the cohesive failure was mainly within composite resin, this may be related to the induction of microcracks during the cutting procedures.[25] The predominant adhesive failure could be explained by the reality that the microtensile bond strength test estimates a small interfacial bonding zone. Furthermore, the strong bond strength of resin cement with the composite resin than with the ceramic.[38]

V. Conclusion

Within the limitations of this in-vitro study, it was concluded that;

1) The bond strength was influenced by the different surface treatment methods and aging process.
2) The hot chemical etching treatment recorded the highest bond strength after aging.
3) Aging process had an important role in the degradation of the bond strength between resin cement and zirconia ceramic.

Acknowledgements

This research was supported by Mansoura University, Mansoura, Egypt.

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Amal Abdelsamad Sakrana. "Microtensile Bond Strength of Zirconia Ceramics with Different Surface Treatments." IOSR Journal of Dental and Medical Sciences (IOSR-JDMS), vol. 18, no. 3, 2019, pp 80-85.