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EFFECT OF AGING ON THE SHEAR BOND STRENGTH OF ZIRCONIA CERAMIC WITH DIFFERENT SURFACE TREATMENTS

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ABSTRACT

Statement of the problem: Zirconia is an acid etching resistant ceramic and may cause difficulty in obtaining a safe bond with it. The aim of this study is to evaluate the effect of aging on the shear bond strength of zirconia ceramic after being subjected to different surface treatments.

Materials & Methods: Forty zirconia samples were constructed in the form of discs by copy milling. All the samples were 5mm in diameter and 1mm in thickness. The samples were divided into four groups according to the type of surface treatment they were subjected to, group1: control, group 2: subjected to etching using hydrofluoric acid 9% , group 3: sandblasting using 50μm Al_2O_3 particles and group 4 : silica coating using 50μm Al_2O_3 and 50 μm SiO_2 particles. The samples were cemented on the occlusal surface of molar teeth using Rely X^{TM} U200. Each group was further subdivided into two subgroups according to the type of test: shear bond strength before aging, shear bond strength after aging. The samples of the second subgroups were thermocycled (5– 55 °C/5,000 cycles) as aging procedure. Shear bond strength was measured. Data were tabulated and statistically analyzed using one-way analysis of variance (ANOVA).

Result: Results showed that the highest shear bond strength before aging was recorded by the silica coated samples while the lowest shear bond strength was recorded with the hydrofluoric acid etched samples. There was no statistical significant difference between all the tested different surface treated samples regarding shear bond strength before aging except the silica coated samples. After aging, the most affected samples with decrease in the shear bond strength were the control samples while there was no significant difference for the tested surface treatments.

Conclusion: Silica coating recorded the highest shear bond strength. Hydrofluoric acid etching and sandblasting are not reliable methods of surface treatment for production of safe and strong bond with zirconia. Aging has a significant effect on the control zirconia samples which showed decrease in the shear bond strength.

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INTRODUCTION

The most important factors that may influence the final choice of dental materials are the strength and esthetics of the prosthesis. Ceramo-metallic fixed partial denture is considered as the gold stand material. However, the great demand to esthetics as well as biocompatibility of prosthetic materials led to the development of new products. Nowadays, ceramic prostheses are replacing ceramo-metallic restorations. A variety of ceramic systems are developed for single crowns or fixed dental prosthesis with an excellent esthetic outcome. Traditional ceramics (glass reinforced and feldspathic ceramics) have some drawbacks as regards to strength. Transformation toughened zirconia ceramics is a successful alternative in different clinical situations because of its high flexural strength. (1)

Zirconia can be used as an alternative material to metal in situations with high loading forces in the posterior region. The high flexural strength of zirconia materials as well as its biocompatibility and esthetics enable it to be used in different situations as posts and cores, dental implants, orthodontic brackets and fixed-partial denture.(2)

Zirconia is a polycrystalline non-etchable material. Adhesion is still an issue since a very low and unstable resin bond is promoted when the Y-TZP ceramic is untreated or receives primer application only. Clinically, debonding of zirconia-based crowns; is a type of restorative failure. Searching for surface treatments that improve resin adhesion to zirconia has increased. Various treatment methods are available as hydrofluoric acid etching, sandblasting and tribochemical silica coating. (3)

Conventional methods of adhesive cementation which include acid etching of the ceramic surface with hydrofluoric acid and further salinization are not efficient for zirconia ceramics due to lack of silica and glass phase **(4)**. But Chaiyabutr et al (2008) (5) stated that hydrofluoric acid etching of zirconia ceramic produced a significant difference in the surface roughness. Clinicians have used airborne particle abrasion with bigger particle size to produce higher surface roughness and hence higher mechanical retention (6). Sandblasting is considered to create surface roughness and irregularities, increase the surface wettability of the ceramic surface. Also it causes cleaning of the substrate's surface thus allowing the resin cement to flow into the surface (7).

Various luting agents can be used with zirconia ceramics as zinc phosphate and modified glass ionomer and resin cements. The use of resin cement is advantageous over other types due to good marginal seal, good retention and improvement of fracture resistance. However, it is difficult to obtain a safe bond between the resin cement and this strong ceramic. Derand et al (2005) (8) reported that aging of ceramics bonded using composite luting cements may affect the bond strength.

The purpose of this research was to study the effect of different surface treatments as well as, aging effect on the shear bond strength of zirconia and resin cement.

The hypothesis of this study was that different types of surface treatment as well as aging would affect bond strength between the zirconia ceramic and resin cement.

MATERIALS AND METHODS

Samples preparation

A split teflon mold with a diameter of 5mm and thickness of 1mm was constructed. It was filled with composite resin (VOCO Grandio C E 0482,Cuxhaven, Germany) of shade A3. It was light cured for 20 seconds using Optilux 400 system (3M ESPE, Seefeld, Germany) to produce a resin pattern. The produced resin pattern was used to produce forty zirconia samples (Prettau, Zirkonzahn, Gais, Italy) in the form of discs using a copy milling machine (Zirkonzhan, Milano, Italy). The resin pattern was fixed at one side of the milling table and the zirconia block at the other side of the table. Pre-sintered zirconium disc was trimmed using Milling Bur 4 L and Milling Bur 3 L (Zirkonzahn, Roseville, Australia). The samples were polished using zirconia polishers (Zirkonzhan, Gais, Italy) and any sharp edges were removed using silicon carbide papers (Microdent, 3R Ind, Com. Brazil). Then, sintering procedure was performed according to manufacturer`s instructions. Discs were put under the infrared predying lamp (Zirkonzahn, Gais, Italy). The firing cycle was carried out at DeguDent sintering machine (DeguDent GmbH Hanau-Wolfgang, Germany) for 5 hours. Then, the thickness of the samples was assessed using a digital micrometer (Praecimeter S, 0.01 mm; Renfert Gmb H, Hilzingen, Germany). After finishing, all the discs were subjected to ultrasonic cleaning (Cavitron, Dentsply Intl, York, Pa, USA) on both sides in distilled water to remove any residues on the surface and air dried.

Freshly extracted upper molar teeth were collected from Periodontic Clinics. Teeth with approximate dimensional similarity were selected. The teeth were selected free from caries or any developmental defect. The teeth underwent ultrasonic cleaning to remove debris and then stored in distilled water till use.

The roots of the teeth were notched to increase retention when embedded in acrylic resin. The teeth were then attached with a sticky wax (Dentsply Sticky Wax; Dentsply Intl, York, PA, USA) to a paralleling device (Paraskop, Bego, Bremen, Germany) on a vertically prepared surface. The teeth were then lowered in a metal ring and positioned in the center of the ring with the cemento enamel junction 3 mm above the top of the metal mounting ring which was filled with auto- polymerizing acrylic resin (Imicryl, Konya, Turkey).

Following the polymerization of acrylic resin the paralleling device rod was detached. The teeth were stored in distilled water all the time after mounting. Occlusal surfaces of the teeth were prepared by removal of occlusal enamel using a dental trimmer to produce flat occlusal surface. The occlusal surface of the teeth were evaluated using a stereomicroscope to evaluate the absence of enamel and pulp tissue on the occlusal surface.

Surface treatment of zirconia discs

The ceramic samples were divided randomly into four groups (10 samples each). The first group was not subjected to any surface treatment and was used as control. The second group was subjected to acid etching using hydroflouric acid 9% (Ultra dent, 3M ESPE, Seefeld, Germany) for 60 seconds, the gel was rinsed off with water for 20 seconds then dried with oil free compressed air (9,10). The third group was subjected to sandblasting using 50μm Al_2O_3 at 80 psi for 5 seconds using sandblasting machine (Renfert GmbH- 78247 Hilzingen- Germany), the samples were mounted in a special pen at a distance of 10 mm between the surface of the sample and the blasting tip. (11) The fourth group was silica coated. The samples were first blasted with 50μm Al_2O_3 particles at 2-3 bars air pressure from a distance of 10 mm for 30 seconds. The samples were then ultrasonically cleaned for 10 minutes in a water bath, then dried. Finally, the samples were blasted with a mixture of 50μm Al_2O_3 and 50 μm SiO_2 particles (Rolloblast 50 μm , Renfert, Germany) at 2-8 bar pressure from a distance of 10 mm for 20 seconds(12)

Zirconia ceramic discs were bonded to the teeth using the following procedure. Silane coupling agent (Ultra dent, 3 MESPe, Seefeld, Germany) was applied using a micro-brush (Epic, Shangahi, China) on the fitting surface of zirconia discs for 90 seconds then dried. Then a dispensed quantity of base and catalyst pastes from resin cement clicker (Rely X^{TM} U200, 3M ESPE, Neuss, Germany) was placed on a mixing pad. Base and catalyst pastes were mixed to a homogenous paste within 20 seconds using a cement mixing spatula (Miltex, INOX, Germany). The homogenous mixed paste was painted on the bonding surface of each disc and then seated on the prepared teeth. Light polymerization was done for 2 seconds firstly, to facilitate removal of the excess cement which was removed using a micro-brush. Then samples then were placed under constant static load (5kg) using the loading device (Model 3345, Industrial products, MA, USA). Finally, light polymerization was done for 40 seconds per surface using Optilux 400 system. Cementation procedures were done by the same operator.

Samples from each group were then randomly divided into two subgroups (5 samples each) according to whether they will be subjected to aging before the shear bond strength test or not.

Shear bond strength test before aging:

The samples of the first subgroup from each group which were used for measuring shear bond strength before aging. Prior to testing, all samples were stored in distilled water for 24 hours. Then, they were mounted in the jig of the universal testing machine (Instron Industrial Products, Norwood, MA, USA) and the load was applied at the adhesive interface where the machine was moved at a crosshead speed of 1mm /min for all the samples until failure had occurred. The bond strength (S) values (expressed in MPa) were calculated using the formula: $S=L/A$[13,14]

where: L: is the load at failure (in N).

A : is the adhesive area (in mm^2) measured using a digital caliper (71170057, Mitutoyo,Tokyo, Japan). Figure (1)

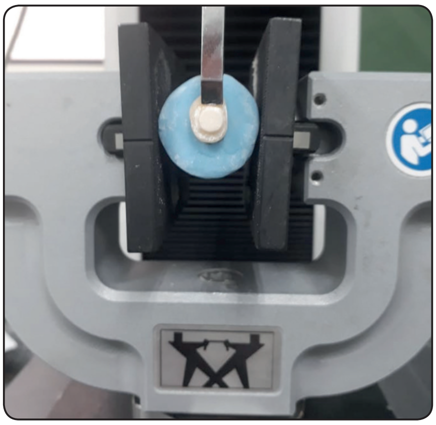

Fig. (1): The tested sample attached to universal testing machine

Aging process:

The samples of the second subgroup from each group which were used for measuring shear bond strength after aging were thermocycled (5– 55 °C/5,000 cycles) (15) using a thermocycling apparatus (Proto-tech: Version 2.1a, Portland, Ore, USA), then the samples were removed, dried and subjected to measuring shear bond strength test after aging following the same parameters as before aging.

Calculations of samples size were performed using R statistical package (version 3.3.1). The proper sample size was detected using the T test power calculation. Estimations of means differences and standard deviations were made according to Derand (2005)(8) based on shear bond strength which is the prime outcome for this study. The sample size calculation was based on the mean difference of 40 samples. Results showed that a total sample size of twenty samples will be adequate to detect a mean difference between the tested groups of 40 samples with a power of 90% and a two sided significance level of 5% with equal allocation to two arms (10 samples in each group).

Data was collected, calculated, tabulated and statistically analyzed using one-way ANOVA Test. Then, a Tukey test was performed to determine significant differences between the tested groups using a confidence level of 0.05 ($p < 0.05$).

RESULTS

Means and standard deviations of the shear bond strength values for the tested groups are presented in table (1) and figure (2). A one way ANOVA Test was used to determine significant difference between the tested groups ($p < 0.05$). Tukey test for multiple comparisons of mean at ($p < 0.05$) was performed following the one-way analysis of variance. Results showed that before aging, the silica coated samples recorded the highest shear bond strength values (5.71 MPa) followed by the control subgroup (2.91 MPa), then the sandblasted subgroup (2.74 MPa). The hydrofluoric acid etched subgroup gave the least shear bond strength (2.69 MPa). There was a statistical significant difference between silica-coated subgroup and the other 3 subgroups. No statistically significant difference was found between the control subgroup and both the hydrofluoric etching subgroup and the sandblasted subgroup.

As regards to the shear bond strength values recorded for the tested subgroups after aging, all aged subgroups showed decrease in the shear bond strength than before aging. There was no statistical significant difference regarding the tested surface treatments before and after aging. The control group, showed a significant difference in shear bond strength between before and after aging.

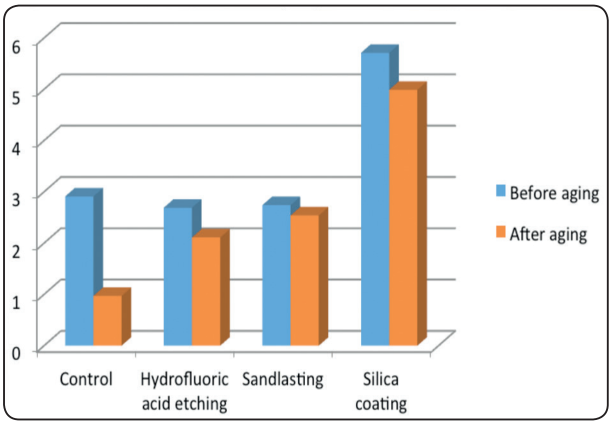

Fig (2): Comparison between shear bond strength of the tested groups before and after aging in MPa

DISCUSSION

This current study evaluates the effect of different surface treatments on the shear bond strength between zirconia ceramic and resin cements. Also, the effect of aging on the shear bond strength was studied.

Yttria-stabilized tetragonal zirconia polycrystal is becoming an alternative to other materials for dental applications such as crowns, cores and fixed partial denture ***Raigrodski (2004)*** (16)**.** It is more esthetic than metal alloys and tougher than other ceramic materials ***Chevalier (2006)*** (17)***,*** it has a favorable soft tissue response in terms of health and esthetics ***Josset et al (1999)*** (18)**.**

TABLE (1) Shear bond strength of the tested groups before and after aging in MPa

	Control	Hydrofluoric acid Etching	Sandblasting	Silica coating
Before aging	2.91 [a] (0.69)	2.69 [a] (0.52)	2.74 [a] (0.46)	5.71 [c] (0.51)
After aging	0.97 [b] (0.24)	2.11 [a] (0.56)	2.54 [a] (0.14)	4.99 [c] (0.33)

Same letters denote no statistical significant difference

There is an increase in the use of resin cement in cementation of fixed prosthesis ***EL Mowafy (2001)***(19), since it exhibits good physical, mechanical and adhesive properties compared to conventional cements ***Attar et al (2003)***(**20**).

Discs were prepared using a Zirkonzhan copy milling machine (21)**.** The samples were milled larger than the resin pattern to compensate for sintering shrinkage. The thickness of the samples was assessed using a digital micrometer (11).

Selection of the teeth was done and molars were selected due to the stresses falling on the posterior area (22,23).

The roots of the teeth were notched for retention when embedded in the acrylic resin (24). The teeth were put on the paralleling device using sticky wax to maintain a perpendicular plane between the bonding surface and the shear loading axis of the universal testing machine (11). Stereomicroscope was used to be sure that there was no enamel or pulp tissue on the surface of the dentin (25).

Four different types of surface treatments were done. The control was not subjected to any surface treatment. The second group was subjected to etching using hydrofluoric acid gel 9%. The third group was subjected to sandblasting where the surface was conditioned using AL_2O_3 particles (11). The fourth group was silica coated where the surface was blasted with a mixture of Al_2O_3 and SiO_2 particles.

Unicem currently marketed as Rely X U200 which was the first cement to contain a methacrylated phosphoric ester as a functional monomer, was the most promising material in terms of adhesion to zirconia (**26**).

With the increase in the use of monolithic complete contour crowns and fixed partial dentures the effect of the long term aging must be determined ***Beuer et al (2012)*** (**27**)**.** The contact of zirconia with humidity causes the transformation of the tetragonal phase to monoclinic one which occurs on the surface of zirconia and negatively affects its mechanical properties. (28-30). The samples were aged by subjecting them to thermocycling (15,31,32), The aging effect induced by thermocycling can occur by repetitive contraction/expansion stresses generated by different thermal coefficient of the restorative materials or by hydrolysis of the interfacial components(15) .

Shear stresses are believed to be major stresses involved in vivo bonding failures of restorative materials, ***Soderholm (1991)***(33) ***Sano et al (1994)***(34) ***and Swift et al (1995)*** (35).

The results of this study showed that silica coated zirconia ceramics gave rise to the highest shear bond strength compared to the other surface treatments tested as well as the control group, as there was a statistical significant difference between the silica-coated subgroups and the other tested subgroups. This may be attributed to that blasting the ceramic with Al_20_3 and SiO_2 combines micromechanical retention produced by airborne-particle abrasion and chemical bonding resulting from silica coating. Due to the absence of silica in Y-TZP, silica-coating techniques would create the chemical bonding provided by silanization.(36)

The results of the current study showed that there was no effect of the surface treatment on the shear bond strength between the control, and the sandblasted and hydrofluoric acid etched subgroups before aging. (37) This also in agreement with ***Manicone et al (2007)***(38), ***Tanaka et al (2008)*** (36)***, de Oyague et al (2009)*** (39) and who reported that surface treatment is not necessary for bonding to zirconia ceramic and the use of a phosphate monomer containing luting system is recommended for bonding to zirconia. This result in controversy with ***wolfort et al (2007)*** (40) and ***Ozcan and Kerkdijk (2008)*** (41) where they stated that air borne particle abrasion enhance bonding to zirconia ceramic.

In regards to sandblasting of zirconia ceramic and its effect on the shear bond strength, this result is in agreement with ***Usumez et al (2013)*** (42) who stated that sandblasting did not increase the bond strength of zirconia comparable to the control group. Also this result is in agreement with ***Kern and Wenger (1998)*** (43)***, Wenger and Kern (2000)*** (44) ***and Derand P and Derand T (2000)*** (45), ***Atsu SS et al (2006)*** (46)***, Ural et al (2010)*** (47). This may be attributed to the fact that air borne particle abrasion can only produce mild coarsening of the zirconia surface which may produce weak bond strength. (48)

Also, hydrofluoric acid etching of zirconia ceramic, in agreement with ***Awliya et al (1998)*** (49), ***Piwowarczyk et al (2005)*** (50), ***Della Bona A et al(2007)*** *(51)****, Donassollo TA et al(2009)***(52) ***Ural et al (2010)*** (47) ***and Mattiello RDL et al (2013)***(53) did not show increase in the bond strength compared to the control group. This may be due to the fact that hydrofluoric acid etching does not produce any change in arithmetic roughness of ZrO_2(9). Also it may be due to that there was a negligible effect of the hydrofluoric acid etching on the ZrO_2 surface due to the absence of glassy matrix resulting in low bond strength values.

Results after aging showed that the control subgroup was the most affected one with significant decrease in the shear bond strength.

This result was in agreement with ***Wolfort et al(2006)***(40) who stated that the bond was not stable over time on speciemens with original surface conditions and debonded spontaneously during aging in contrast to the air abraded group which maintained a durable bond strength over the aging time. This result is also in agreement with ***Inokoshi et al (2011)*** (54) who found that untreated zirconia degraded faster than the surface treated zirconia.

The hypothesis of this research was partially accepted as only silica coating affected shear bond strength, while there was no significant difference between the other 2 surface treatments and the control subgroup, as regards samples tested before thermocycling. Also the hypothesis was partially accepted for samples tested after aging as only the control subgroup showed significant difference , while the was no significant difference between aged and non-aged samples for the tested surface treatments.

CONCLUSIONS

(1) Silica-coating zirconia ceramics yielded the highest shear bond strength compared to the control samples and the other tested surface treatments.

(2) Hydroflouric acid etching and sandblasting as methods of surface treatment of zirconia ceramic are not reliable methods for production of safe and strong bond with zirconia.

(3) Aging has a significant effect on the control zirconia samples which showed decrease in the shear bond strength. While this effect, was not statistically significant in case of surface treated zirconia samples.

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