

# EFFECT OF DIFFERENT SURFACE TREATMENTS ON BONDING OF ULTRA-TRANSLUCENT ZIRCONIA

Dina M. Nasr<sup>1\*</sup> MSc, Samir A. Koheil<sup>2</sup> PhD, Waleed A. El Mahy<sup>2</sup> PhD.

## ABSTRACT

**INTRODUCTION:** Introduction of ultra-translucent zirconia led to increase its indications for minimally invasive aesthetic restorations, therefore, it is important to test its adhesive potentiality.

**OBJECTIVES:** Investigation of shear bond strength (SBS) of ultra-translucent zirconia to enamel after different surface treatments and examination of their effect on surface topography.

**MATERIALS AND METHODS:** Thirty six ultra-translucent zirconia discs, ( $\text{\O}5\text{mm}\times 3\text{mm}$  height) were divided into three groups ( $n=12$ ): Control (C): as-sintered discs, Sandblasted discs (SB): discs were sandblasted by  $110\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles for 10 seconds, at 2.5 bar pressure, from 10mm distance, and Etched discs (ZE): discs were dipped into Zircos-E etching solution (BIO DEN CO, Korea) for thirty minutes in an ultrasonic cleaner. The surface topography of two randomly selected discs from each group were examined by scanning electron microscopy (SEM) for descriptive analysis, and the remaining discs were bonded to enamel surfaces of human central incisors for performing the SBS test. Statistical analysis of SBS results was done by Kruskal Wallis test and post hoc test with statistically significant results at ( $p<0.05$ ).

**RESULTS:** Morphological examination of (C) showed closely connected grains, (SB) showed a number of superficial irregularities, and shallow scratches, whereas (ZE) showed gross discontinuity of grain structure. The highest SBS value was recorded by (SB) ( $19.01\pm 2.00$  MPa) with insignificant difference from (ZE) ( $16.73\pm 2.13$  MPa), and significant difference from (C) with the lowest SBS value ( $8.69\pm 1.95$  MPa).

**Conclusions:** Etching of ultra-translucent zirconia using (Zircos-E) is a promising technique for improvement of resin bonding in comparison to control and sandblasted groups.

**KEYWORDS:** Shear bond strength, Ultra-translucent zirconia, sandblasting, Zircos-E solution.

**RUNNING TITLE:** Bonding of Ultra-translucent Zirconia to Enamel.

1. Assistant lecturer at Conservative Dentistry Department, Faculty of Dentistry, Alexandria University.
2. Professor of Conservative Dentistry, Conservative Dentistry Department, Faculty of Dentistry, Alexandria University.

\* Corresponding Author:

E-mail: denanasr72@gmail.com

## INTRODUCTION

The high strength ceramics based on conventional tetragonal zirconia polycrystal (TZP) with outstanding mechanical properties (flexural strength  $\approx 1200$  MPa); had an unpleasant white colour and lack of translucency that made them difficult to replicate the look of natural teeth (1). As a solution for the high opacity of TZP; feldspathic porcelain was veneered on TZP core to match the colour and translucency of natural teeth, however, high rates of chipping have been reported for all-ceramic restorations with TZP core in comparison to porcelain fused to metal restorations (2).

To overcome this problem, monolithic (fully anatomic) TZP restorations, were introduced for making a multiunit anterior monolithic fixed restorations without the increased risk of porcelain fracture or reduced aesthetic properties of the core material (3). However, their low translucency limits their clinical application in the anterior aesthetic zone (4).

The monolithic zirconia had passed through modifications of the composition by decreasing the alumina content that had a different refractive index ( $n = 1.76$ ) from that of zirconia ( $n = 2.21$ ) (4), also, increasing the yttria content (up to 9.42 wt% as compared to approximately 5.15 wt% for conventional zirconia), led to a subsequent increase in the amount of cubic phase with its larger size that had a higher translucency level (5). In addition to this, the increase of the grain size with a respectable density and diminished pores in the polycrystalline structure was detected by increasing the sintering duration and temperature (4).

The most recent high translucent zirconia is the cubic ultra-translucent (UT) zirconia (with increased cubic phase content and lower flexural strength of  $\approx 500- 800$  MPa) (6). The increase of the translucency of zirconia without significantly losing their fracture resistance led to advancement in its use

for the aesthetic minimally invasive monolithic zirconia restorations, including veneers and ultra-thin veneers (7).

Besides the upgrading of translucency, the success of zirconia-based all-ceramic restorations necessitate a good adhesion between zirconia and luting cement. However, a major impairment to its use is related to its nearly inert status since it is non-polar with a very dense and homogeneous structure after sintering (8). Also, there is no intrinsic glass content in the matrix of zirconia so that zirconia base structures cannot be etched with commonly used concentrations of hydrofluoric acid (HF), for enhancement of the surface roughness (9).

There are multiple techniques that had been investigated in multiple systematic reviews, to overcome the obstacle of obtaining a reliable long lasting bond strength to zirconia in in-vitro studies (9-13). Based on the results of these systematic reviews, improved adhesion could be obtained after doing a combined mechanical and chemical surface treatments of zirconia; where the air particle abrasion with aluminium oxide particles  $Al_2O_3$  with different size range (25 to 250  $\mu m$ ) was included in most of research protocols and it was considered a reference method, (10). This technique created a large surface area and mechanical undercuts for adhesion (14). This was accompanied with the chemical treatment of the surface with e.g.: an MDP (methacryloyloxydecyl dihydrogen phosphate) ceramic coating material and the usage of a MDP-based adhesive resin cements (13).

Recently, a material was introduced for chemical etching of the zirconia (Zircos-E etching agent, liquid base etching solution, BIO DEN CO, Seoul, Korea). It is a mixture of Hydrofluoric acid (HF), hydrochloric acid (HCl), Sulfuric acid ( $H_2SO_4$ ), Nitric acid ( $HNO_3$ ) and Phosphoric acid ( $H_3PO_4$ ). It roughens zirconia surface to increase the surface area through preconditioning; it could enhance interfacial adhesion and eventually increase the bond strength between the zirconia and teeth (15,16).

The high-strength ceramic materials with improved translucency are becoming more commonly used with increased indications, therefore, it is of a great interest to evaluate the adhesive potentials of these materials (17).

Thus, the null hypothesis of this study was that the different surface treatments of ultra-translucent zirconia would have similar effects on both surface topography of the material and shear bond strength of the material to tooth enamel.

## MATERIALS AND METHODS

### 2.1 Ultra-translucent zirconia discs preparation

Thirty-six disc shaped specimens ( $N=36$ ), ( $\varnothing$  5mm  $\times$  3mm height) of ultra-translucent multi-layered (UTML) katana zirconia (Kurary, Noritake Dental Inc, Okayama, Japan), (composition is listed in Table.1), were prepared by a CAD/CAM milling machine, (Roland DWX-50 CAD/CAM Dental Milling Machine, Hamamatsu, Japan). The pre-sintered milled discs were sintered by a sintering furnace (Mihm-Vogt high-temperature furnace. Mihm-Vogt GmbH & Co, Stutensee-Blankenloch, Germany) at 1550 °C/ 2822 °F with a heating rate of  $\pm 10^\circ C$  (18 °F)/min and two hours holding time according to the manufacturer's instruction (18).

The discs were checked regarding the dimensions by a digital caliper (Titan Electronic Digital Caliper, Pennsylvania, USA). The ones that were not accurate regarding the diameter by more than  $\pm 0.02$  mm were excluded from the study.

All discs were rinsed with tap water for 1 min, dipped in a 90% ethanol alcohol bath and ultrasonically cleaned using (ANGEL POS Ultra-sonic cleaner, cd4820, 160W, Burnaby, Canada) for 30 min and gently air-dried (19).

The cleaned discs were divided randomly into three groups ( $n = 12$ ) according to the applied surface treatments into: **Group (1):** Control (as-sintered discs), (C), without any surface treatment, (5). **Group (2):** Sandblasted discs (SB), that were sandblasted with 110  $\mu m$   $Al_2O_3$  (OxidoAluminio, Protechno, Girona, Spain) for 10 seconds at a pressure of 2.5 bar from 10 mm distance (20). **Group (3):** Etched discs (ZE), that were etched by Zircos-E etching solution (BIO DEN CO, Seoul, Korea) for thirty minutes in an ultra-sonic cleaner, (composition is listed in Table. 1), (personal protection measures using the mask, eye glasses and heavy-duty gloves were a must at the time of insertion and removal of the specimens from the etching solution container), the discs were dipped in Zircos-E etching solution container and it was closed accurately and placed in an ultra-sonic cleaner, using normal mode up to temperature (40° C /104° F) (ANGEL POS Ultra-sonic cleaner, cd4860, 300W, Burnaby, Canada) and left undisturbed for exactly 30 minutes, subsequently, the discs were removed and rinsed in cold running water for 2 minutes, this was followed by steam cleaning and annealing process at a temperature (1150° C/2102° F) with thirty minutes holding time at this temperature, according to the manufacturer's instruction (16). This annealing process is a method to relieve the residual stresses and to remove residual substances (15).

### 2.2 Surface topography examination

The SEM was used for the examination of the surface topography of two randomly selected disc specimens from each group. The disc specimens were ultrasonically cleaned in 90% ethanol for 10 minutes and gently air-dried, subsequently, they were mounted on metallic stubs, gold sputter-coated by (JEOL Fine Coat Ion-sputter JFC-1100, JEOL, Tokyo, Japan) and examined by (JEOL JSM-6360LA SEM, Tokyo, Japan) at (5,000 x) magnification to examine the changes in the surface topography (19).

### 2.3 Shear bond strength test (SBS) of Ultra-translucent zirconia to enamel:

#### 2.3.1 Preparation of the natural central incisor teeth:

The remaining ten discs from each group (C, SB, ZE) were bonded to the enamel surface of thirty natural upper central incisors that were extracted due to periodontal involvement, from out-patients attending the Oral surgery department clinics, Faculty of Dentistry, Alexandria University. This in-vitro experimental study was approved by the ethics committee in the Faculty of Dentistry, Alexandria University. The teeth used for this test were free of caries, cracks, fluorosis or previous endodontic treatments. The teeth were stored in normal saline and external debris were removed using a hand scaler (19). A diamond disc (D-201 Diamond Disc, 0.2mm flexible disc, blue dolphin products, California, USA) was used on a low speed micromotor to flatten the incisal two thirds of

the labial surface of the teeth to be adequate for bonding to the flat surface of the zirconia discs, conditioned that the preparation was still in enamel, the root part of each tooth was sectioned 1 mm apical to the cement-enamel junction with the diamond disc to obtain the crown part. Each crown was seated with the labial surface up in self-curing acrylic resin that was placed in a copper metallic split mold to form a cylindrical specimen, (diameter: 15 mm; length: 25 mm). After solidification of the self-curing resin, the labial surfaces of the crowns were polished with silicon carbide (SiC) abrasive paper (gradually from grit # 800, 1000 to 2000) to remove any remnants of acrylic resin and expose enamel surface (19).

### 2.3.2 Cementation Procedure and Thermocycling Test

The thirty discs from the three groups (C, SB, and ZE) were cleaned with 37% phosphoric acid gel (Meta etchant, Meta Biomed Co., Korea) for 1 minute, washed and air dried. The cleaned discs were dipped in 3 different containers filled with 90% alcohol in an ultrasonic bath for 10 minutes, each belonging to its group, after that they were removed and dried (21). The UTML zirconia discs from the three groups were randomly assigned to the previously prepared central incisors, to be cemented to the teeth using the Panavia V5 resin cement kit (Kuraray, Noritake Dental Inc, Japan), (compositions of the used materials are listed in Table.1).

Each disc was primed by the CLEARFIL CERAMIC PRIMER PLUS on only one surface of each treated disc specified for bonding to the tooth structure, and it was blown dry with water and oil free air (22), while it was held by a metallic forceps for its fixation during the drying step. The enamel surfaces of the teeth were etched by phosphoric acid (K-ETCHANT gel, 35% etching gel) for 15 seconds, washed and blown dry with mild air. The etched enamel surfaces were primed by the Panavia V5 tooth primer, that was spread on the enamel surface and left for 20 sec, subsequently, it was blown dry with mild air(23).

The Panavia V5 resin cement pastes A and B were mixed by the auto mixing tip and applied to the treated surface of the discs and teeth on the flattened area of the incisal two thirds of their labial surface; this was followed by the placement of the primed disc with the primed surface facing toward the tooth. Each tooth with its corresponding bonded UTML zirconia disc were compressed by a customized apparatus that produced a constant load of 2 kg, (as shown in Fig. 1). Excess resin cement was wiped off with a micro brush while the specimens were under the constant load (17,23).

The specimens were light-cured with a halogen light cure lamp (Chromalux- E, Mega-physic Dental, GmbH & Co, KG, Germany, with a wavelength :400-500nm and light intensity 600-800 mW/cm<sup>2</sup>) for 40 seconds from each side to ensure optimal polymerization(the additional chemical curing mechanism of the material lasted for 3 minutes). The constant load was removed and excess resin cement was removed carefully using a dental scalpel (24).All of the bonded specimens were stored in distilled water for 24 hours (19),then placed in a thermocycling apparatus (Designed and fabricated by the Dental Materials Department, Faculty of Dentistry, Alexandria University) using distilled water for 10,000 cycles between 5° and 55° C which is clinically equivalent to

approximately one year of clinical service (25). The dwell time at each temperature was 30 seconds and the transfer time was 2 seconds (ISO 10477-1996) (19,26).

### 2.3.3 Shear Bond Strength Test and Fracture Mode Examination:

Each bonded specimen was loaded onto a universal testing machine (Autograph AG-I, SHIMADZU Corporation, Kyoto, Japan). The shear bond strength test was performed according to the guidelines specified in the ISO/TS 11405-2003, The shear force was applied parallel to the interface of the bonding surfaces at a speed of 0.5 mm/min until bonding failed, (Fig. 2)(27).The shear force was recorded automatically at the point of failure, with the taken values indicating the Load force by (N). The shear bond strength was calculated by dividing the force by the interface area, using the following equation: [Shear bond strength (MPa) = Load (N)/area (mm<sup>2</sup>)] automatically using a computer software program (Trapezium2, SHIMADZU Corporation, Kyoto, Japan).

### 2.3.4 The failure modes

After debonding, the fractured interfaces of the specimens were examined using a stereo-microscope at (30x) (Clympus JZ 1145 TR, Japan), to determine the failure modes as follows: ZRM: (zirconia resin mode) zirconia surface is free of resin cement, ERM: (enamel resin mode) enamel surface is free of resin cement, MRM: (mixed resin mode) resin cement remnants are seen on both enamel and zirconia surfaces, EFM: (enamel fracture mode) enamel fractured part is adherent to zirconia surface, ZFM: (zirconia fracture mode) zirconia surface has a deficient part, and CFM: (cohesive failure mode) adhered resin cement covers the whole surface of both the zirconia and enamel (19).

### 2.3.5 Statistical analysis

The scanning electron micrographs of surface examination by SEM, were described by descriptive analysis, whereas, the shear bond strength results were collected and entered to the computer using SPSS (Statistical Package for Social Science) program for statistical analysis (ver 21). Data were entered as numerical data and described using minimum, maximum, mean, standard deviation and 95% CI of the mean, median and inter-quartile range. Comparisons were carried out between the three studied groups using Kruskal-Wallis test. Post-hoc pair-wise comparisons were used when Kruskal-Wallis test was significant. Box and Whiskers plot, was used accordingly. Regarding the failure modes the specimens were categorized to the different failure modes and then calculated as a percentage from the total number of specimens at each group for calculation of their probability percentage.

## RESULTS

### 3.1 Surface examination by Scanning Electron Microscope (SEM)

Morphological examination of the disc specimens at (5,000x magnification) showed that, the (C) specimens showed the closely connected grains in the lattice structure, (Fig.3 a). The (SB) specimens showed superficial irregularities and shallow

scratches, accompanied with the presence of some impinged abrasive particles on the ultra-translucent zirconia surface, (Fig. 3 b), on the other hand, the (ZE) specimens showed different degrees of roughness with gross discontinuity of grain structure,(Fig. 3 c).

**3.2 The shear bond strength test results (SBS)**

Mean shear bond strength values (MPa), standard deviation, median, and Inter-quartile range, for each group are listed in (Table: 2, Fig. 4). **Kruskal Wallis Test** revealed a significant difference between the groups [ $H = 21.461, p=0.000^*$ ].

**Pairwise comparison** was performed between each two groups using **Post Hoc Test**, the statistical comparison between groups (C, SB), showed that (SB) resulted in a higher SBS than (C) and the difference is statistically significant ( $p=0.000^*$ ), also, statistical comparison between (C& ZE), showed that (ZE) resulted in a higher SBS than (C), and the difference is statistically significant ( $p = 0.006^*$ ), whereas, statistical comparison between subgroups (SB& ZE) showed that (SB) resulted in a higher SBS than subgroup (ZE), but the difference is statistically not significant at ( $p = 0.443$ ).

**3.2.1 The failure modes**

The (C) group showed nine specimens with ZRM at 90% probability, and the tenth specimen showed an MRM at 10% probability within the group. The (SB) group showed four specimens with ZRM at 40% probability, three specimens with MRM at 30% probability, two specimens showed CFM at 20% probability, and the tenth specimen showed EFM. The (ZE) group showed three specimens with ZRM at 30% probability, four specimens with MRM at 40% probability, and three specimens with CFM at 30% probability within the etched specimens. Neither the ERM nor ZFM failure modes were observed in all the examined specimens from the three groups, (Figs. 3 a, b, c, d).

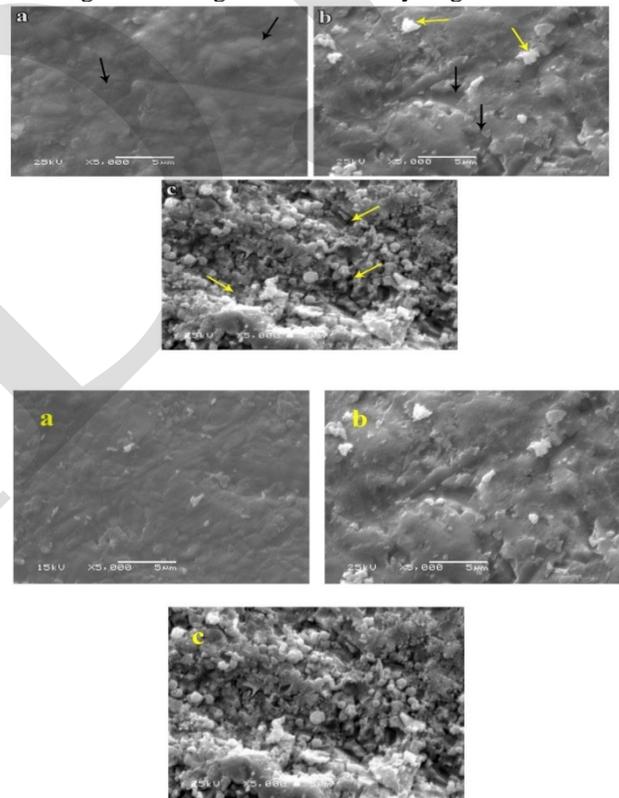
**Figure (1):** The cemented UTML zirconia discs were pressed by customized apparatus that produced a constant load of 2 kg.



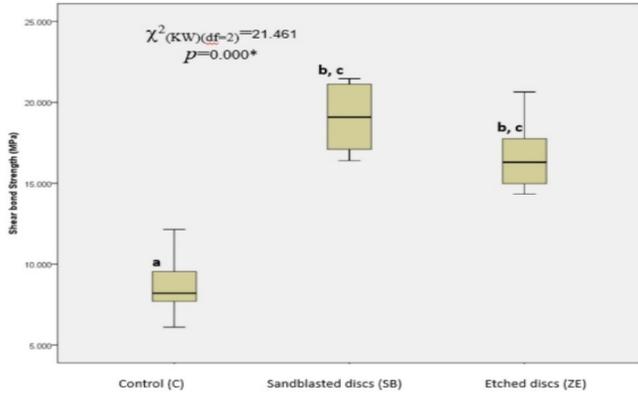
**Figure (2):** The shear force was applied parallel to the interface of the bonding surfaces.



**Figure (3):**SEM micrographs (5000x) showing the surface topography of (a) Control (as-sintered) discs: black arrows are showing the closely connected grains in the lattice structure, (b) Sandblasted discs: black arrows are showing the superficial irregularities and shallow scratches, while the yellow arrows are showing impinged abrasive particles on the surface , and (c) Etched discs: yellow arrows are showing different degrees of roughness with gross discontinuity of grain structure.

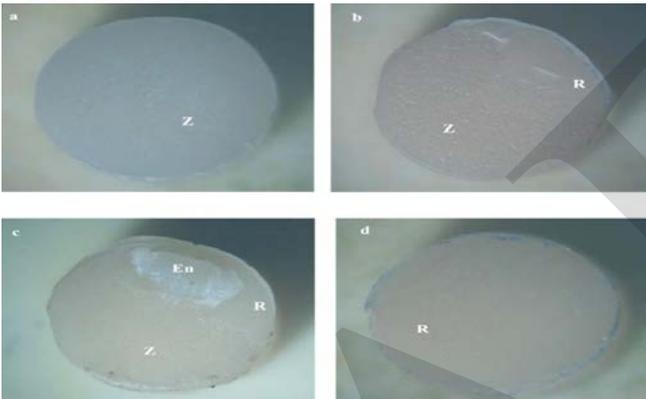


**Figure (4):** Box and whisker graph of Shear bond Strength (MPa) in the studied groups, the thick line in the middle of the box represents the median, the box represents the inter quartile range (from 25<sup>th</sup> to 75<sup>th</sup> percentiles)



**Figure (5):** Stereo-microscopic images showing the different types of failure modes, (30x), (a) ZRM, (b) MRM, (c) EFM, and (d) CFM.

Z: denotes the ultra-translucent zirconia surface, R: denotes the resin cement, and En: denotes a fractured enamel part adhered to ultra-translucent zirconia disc.



**Table (1):** Composition of materials used in the study.

Product	Composition	Manufacturer
Ultra-translucent multi layered Katanzirconia CAD/CAM blanks.(17,22)	ZrO <sub>2</sub> : 89% Y <sub>2</sub> O <sub>3</sub> : 8–11% HfO <sub>2</sub> :2% Al <sub>2</sub> O <sub>3</sub> : 0.16% Other oxides 0–2%	Kuraray Noritake Dental, Kurashiki, Japan
Zircos- E Etching solution (14)	Hydrofluoric acid (HF) (0-25%) Hydrochloric acid (HCl) (0-25%) Sulfuric acid (H <sub>2</sub> SO <sub>4</sub> ) (0-25%) Nitric acid (HNO <sub>3</sub> ) (0-25%) Phosphoric acid (H <sub>3</sub> PO <sub>4</sub> ) (0-25%) Percentages were hidden as a (Trade Secret*)	BIO DEN CO, Seoul, Korea.
1- 2- K-ETCHANT Syringe	35% phosphoric acid aqueous solution, colloidal silica, Polyethyleneglycol, Water, Pigment.	Kuraray Noritake Dental, Kurashiki, Japan
3- Panavia V5 Tooth Primer (40)	10- MDP, HEMA, Hydrophilic aliphatic dimethacrylate, Accelerators, Water.	Kuraray Noritake Dental, Kurashiki, Japan
4- Clearfil Ceramic Primer Plus (22,31)	3-TMSPMA,10- MDP, Ethanol.	Kuraray Noritake Dental, Kurashiki, Japan

**Abbreviations:** ZrO<sub>2</sub>: Zirconium dioxide, HfO<sub>2</sub>: Hafnium dioxide, Y<sub>2</sub>O<sub>3</sub>: Yttrium oxide, Bis-GMA: bisphenol A diglycidyl ether dimethacrylate, TEGDMA: triethyleneglycoldimethacrylate, 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, 3-TMSPMA: 3-trimethoxysilylpropyl methacrylate.

**Table (2):** Mean shear bond strength values (MPa), standard deviations ( $\pm$ ), and statistical results of all groups

Shear bond Strength (MPa)	Group		
	C	SB	ZE
- n	10	10	10
- Min-Max	6.1-12.14	16.4-21.46	14.3-20.65
- Mean $\pm$ Std.	8.69 $\pm$ 1.95	19.01 $\pm$ 2.00	16.73 $\pm$ 2.13
- Deviation (IQR)	8.2 <sup>a</sup> (7.7-9.54)	19.085 <sup>b,c</sup> (17.1-21.12)	16.3 <sup>b,c</sup> (14.98-17.75)
Test of significance	H = 21.461 p=0.000*		

Different lowercase letters indicate significant differences among cements (P<0.05).

n : Number of discs.

KW: Kruskal Wallis Test

Min-Max: Minimum – Maximum

\* : Statistically significant (p<0.05)

CI: Confidence interval

NS: Statistically not significant (p>0.05)

IQR: Inter-quartile range

### DISCUSSION

This study was an attempt to investigate the bonding of ultra-translucent zirconia material to the tooth structure. In this study, the control group (without any surface treatment) was compared to two types of surface treatment modalities that depend on micromechanical roughening of the zirconia surface (the sandblasting and etching procedures) (10). So far, for obtaining the maximum SBS to the high translucent zirconia, it was recommended to use the sandblasting with 110  $\mu$ m particle size from 10 mm distance at 2 or 3 bar pressure, as stated by LE et al (17) and Zhao et al (32), respectively, while Ansari et al (21) found that, thirty minutes etching time by the zirconia etching solution was capable of performing morphological changes of high translucent zirconia and improvement of the bond strength, thus, in this study, both techniques were compared to the control group to investigate their effect on the bond strength of the ultra-translucent zirconia.

In addition to this, the dual cured MDP containing resin cement (Panavia V5 resin cement) was selected for bonding the UTML zirconia discs to the enamel surface of teeth, for formation of a chemical bond between their phosphate groups and the hydroxyl groups on the zirconia surface (31). Our results revealed that, the (SB) group showed the highest SBS value with insignificant difference from (ZE) group, and significant difference from (C) group with the lowest SBS value. The lowest obtained SBS value by the control group disc specimens, might be correlated to performing no treatment to the as-sintered surface, that resulted into inadequate adhesion, this was in agreement with LE et al (17), Inokoshi et al (5), Cheung & Botelho (28), Saade et al (29) and Liu et al (30), where Cheung & Botelho (28) and Liu et al (30) found de-bonded specimens of the as-sintered group that could not even

withstand the aging treatments and got de-bonded before application of SBS test.

The highest SBS values obtained by the sandblasted subgroup in our study, could be related to the uniform existence of superficial irregularities and shallow scratches all over the examined surface, that was related to the effect of hitting the surface by the sharp abrasive particles creating retentive spaces accompanied with the presence of some impinged abrasive particles on the ultra-translucent zirconia surface by the force of blasting pressure that led to increased available surface area for obtaining a micro-mechanical bond with the adhesive resin resulting in the highest shear bond strength results, this was in agreement with, Aung et al (31), Hallmann et al (14), Kern M (11), LE et al (17), Liu et al (30), Thammajaruk et al (13), Tzanakakis et al (10)

and Zhao et al(32), who stated that air-abrasion at a moderate pressure provided a reliable bonding to zirconia based restorations, particularly when combined with phosphate monomer containing primers and/or luting resins as it appeared in this study. However, this was in disagreement with other studies done by Lee et al (2019) (24), Lee & Lee (33), Xie et al (34), and Lee et al., (2015) (20), who found that, the shear bond strength was higher when zirconia was etched with higher concentrations of HF acid or strong acid mixture composed of HNO<sub>3</sub> and HF acids than by using only the air abrasion technique.

Regarding the etched specimens, the etching solution led to a significantly higher SBS results than the control specimens, that could be related to the ability of Zircos -E etching solution to etch the ultra-translucent zirconia with formation of porosities of different shapes and depths, due to its preferential action on the grain boundaries, where the external atoms are more chemically reactive and are more liable to be dissolved earlier than those inside the crystal, with a resultant decrease of the grain size or even dislodgment of the grains themselves, this description was in accordance with the description of etching mechanism of zirconia by Sriamporn et al(35).

The SBS results obtained by Zircos-E etching of the ultra-translucent zirconia, were in agreement with Ansari et al(21), Cho et al (15), and Lee et al (36) who found a significant improvement of SBS results of the etched specimens by Zircos-E etching solution with apparent morphological changes of the surface features of zirconia, that resulted in increased effect on the fully stabilized zirconia than the partially stabilized zirconia due to the presence of high percentage of cubic phase with larger grain size, as stated by Ansari et al(21).

Nevertheless, in this study, the SBS of etched subgroup was less than sandblasted subgroup insignificantly, and this might be attributed to the produced surface porosities by etching solution. However, the high viscosity of the used resin cement in our study with a filler particles that ranged from (0.01–12 µm) at 38% by volume, could prevent its penetration into the nano-porosities created by the etching solution, this coincided with the results obtained by, Sriamporn et al(35), and Cho et al (15), who stated that the less viscous resin cement with no fillers showed better flowability throughout the produced nano-porosities by Zircos-E

etching solution, with the resultant highest SBS in comparison to air abrasion or silicoating surface treatments.

Regarding the failure modes, the control group had a predominantly adhesive failure between zirconia and the resin cement in zirconia resin mode (ZRM), this was in agreement with Cheung & Botelho, and Zhao et al (28,32). Regarding the sandblasted and the etched groups, they showed variable intensities of the different types of failure modes, this was in agreement to Cho et al, and Zandparsa et al (15,37), that could be related to the improved adhesive performance of the resin cement to both the sandblasted or etched ultra-translucent zirconia surface and enamel of the central incisors in mixed resin mode (MRM), or that could even overcome the cohesive strength of resin cement itself in cohesive failure mode (CFM), while, the adhesive performance of the resin cement to the sandblasted ultra-translucent zirconia surface could overcome the cohesive strength of the bonded enamel itself through showing some of the fractured enamel rods adhered to the resin cement remnants on the sandblasted disc specimen in enamel fracture mode (EFM). All of these could postulate that the bond between the sandblasted or the etched ultra-translucent zirconia and resin cement is equivalent to the bond between resin cement and enamel, this was in agreement to Xie et al (38), Zandparsa et al (37). In addition to that, the CFM indicated that, the main source of failure was the structural flaws within the resin itself and not at the interface and this was considered as the best bonding condition that can be achieved, as stated by Hooshmand et al (39).

In this study, the tested surface treatment modalities resulted in significantly higher bond strength values when compared to using the discs in as-sintered condition, with variable effects on the surface topography of the tested specimens, thus, the null hypothesis has been rejected.

## CONCLUSIONS

Sandblasting of ultra-translucent zirconia is considered as an acceptable method for gaining adequate bond strength, also, etching of ultra-translucent zirconia using (Zircos-E) is a promising technique for improvement of zirconia resin bonding in comparison to no treatment or the standard technique of sandblasting.

### Conflict of interest

The authors declare that they have no conflicts of interest.

## REFERENCES

1. Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. *J Prosthodont Res.* 2013;57:236-61.
2. Roediger M, Gersdorff N, Huels A, Rinke S. Prospective Evaluation of Zirconia Posterior Fixed Partial Dentures: four-Year Clinical Results. *Int J Prosthodont.* 2013;26:164-71.
3. Ghodsi S, Jafarian Z. A Review on Translucent Zirconia. *Eur J Prosthodont Restor Dent.* 2018;26:62-74.
4. Zhang Y. Making yttria-stabilized tetragonal zirconia translucent. *Dent Mater.* 2014;30:1195-203.

5. Inokoshi M, Shimizu H, Nozaki K, Takagaki T, Yoshihara K, Nagaoka N, et al. Crystallographic and morphological analysis of sandblasted highly translucent dental zirconia. *Dent Mater.* 2018;34:508-18.
6. Kontonasaki E, Giasimakopoulos P, Rigos AE. Strength and aging resistance of monolithic zirconia: an update to current knowledge. *Jpn Dent Sci Rev.* 2020;56:1-23.
7. Souza R, Barbosa F, Araújo G, Miyashita E, Bottino MA, Melo R, et al. Ultrathin Monolithic Zirconia Veneers: Reality or Future? Report of a Clinical Case and One-year Follow-up. *Oper Dent.* 2018;43:3-11.
8. 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.
9. Luthra R, Kaur P. An insight into current concepts and techniques in resin bonding to high strength ceramics. *Aust Dent J.* 2016;61:163-73.
10. Tzanakakis E-GC, Tzoutzas IG, Koidis PT. Is there a potential for durable adhesion to zirconia restorations? A systematic review. *J Prosthet Dent.* 2016;115:9-19.
11. Kern M. Bonding to oxide ceramics-Laboratory testing versus clinical outcome. *Dent Mater.* 2015;31:4-18.
12. Özcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. *J Adhes Dent.* 2015;17:7-26.
13. Thammajaruk P, Inokoshi M, Chong S, Guazzato M. Bonding of composite cements to zirconia: A systematic review and meta-analysis of in vitro studies. *J Mech Behav Biomed Mater.* 2018;80:258-68.
14. Hallmann L, Ulmer P, Reusser E, Hämmerle CHF. Effect of blasting pressure, abrasive particle size and grade on phase transformation and morphological change of dental zirconia surface. *Surf Coatings Technol.* 2012;206:4293-302.
15. Cho JH, Kim SJ, Shim JS, Lee K-W. Effect of zirconia surface treatment using nitric acid-hydrofluoric acid on the shear bond strengths of resin cements. *J Adv Prosthodont.* 2017;9:77-84.
16. ZIRCOS-E® Etching Solution Technical Guide. BIO DEN CO, Seoul, Korea, 2019. Available at: [http://biodenzircose.com/eng/products\\_zircos-e3.html](http://biodenzircose.com/eng/products_zircos-e3.html)
17. LE M, Larsson C, Papia E. Bond strength between MDP-based cement and translucent zirconia. *Dent Mater J.* 2019;38:480-9.
18. Kuraray Noritake Dental Inc, Katana Zirconia UTML/STML. Tech Guid. 2019. Available at: [https://kuraraydental.com/wp-content/uploads/sds/Guides/katana\\_zirconia\\_utml\\_stml\\_tg.pdf](https://kuraraydental.com/wp-content/uploads/sds/Guides/katana_zirconia_utml_stml_tg.pdf)
19. Lv P, Yang X, Jiang T. Influence of hot-etching surface treatment on zirconia/resin shear bond strength. *Materials (Basel).* 2015;8:8087-96.
20. Lee MH, Son JS, Kim KH, Kwon TY. Improved resin-zirconia bonding by room temperature hydrofluoric acid etching. *Materials (Basel).* 2015;8:850-66.
21. Ansari S, Jahedmanesh N, Cascione D, Zafarnia P, Shah KC, Wu BM, et al. Effects of an etching solution on the adhesive properties and surface microhardness of zirconia dental ceramics. *J Prosthet Dent.* 2018;120:447-53.
22. Yagawa S, Komine F, Fushiki R, Kubochi K, Kimura F, Matsumura H. Effect of priming agents on shear bond strengths of resin-based luting agents to a translucent zirconia material. *J Prosthodont Res.* 2018;62:204-9.
23. Rohr N, Fischer J. Tooth surface treatment strategies for adhesive cementation. *J Adv Prosthodont.* 2017;9:85-92.
24. Lee Y, Oh KC, Kim NH, Moon HS. Evaluation of Zirconia Surfaces after Strong-Acid Etching and Its Effects on the Shear Bond Strength of Dental Resin Cement. *Int J Dent.* 2019;2019:3564275.
25. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent.* 1999;27:89-99.
26. International Standards Organization. Dentistry-Polymer-Based Crown and Bridge Materials; ISO 10477; International Standards Organization (ISO): Geneva: Switzerland; 1996.
27. International Standards Organization (ISO). Technical specification ISO/TS 11405. Dental materials-testing of adhesion to tooth structure. Switzerland; 2003.
28. Cheung GJK, Botelho G. Zirconia Surface Treatments for Resin Bonding. *J Adhes Dent.* 2015;17:5518.
29. Saade J, Skienhe H, Ounsi H, Matinlinna JP, Salameh Z. Effect of different combinations of surface treatment on adhesion of resin composite to zirconia. *Clin Cosmet Investig Dent.* 2019;11:119-29.
30. Liu D, Tsoi JKH, Matinlinna JP, Wong HM. Effects of some chemical surface modifications on resin zirconia adhesion. *J Mech Behav Biomed Mater.* 2015;46:23-30.
31. Aung SSMP, Takagaki T, Lyann SK, Ikeda M, Inokoshi M, Sadr A, et al. Effects of alumina-blasting pressure on the bonding to super/ultra-translucent zirconia. *Dent Mater.* 2019;35:730-9.
32. Zhao P, Yu P, Xiong Y, Yue L, Arola D, Gao S. Does the bond strength of highly translucent zirconia show a different dependence on the airborne-particle abrasion parameters in comparison to conventional zirconia? *J Prosthodont Res.* 2019;pii:S1883-1958:30454-7.
33. Lee JH, Lee CH. Effect of the Surface Treatment Method Using Airborne-Particle Abrasion and Hydrofluoric Acid on the Shear Bond Strength of Resin Cement to Zirconia. *Dent J.* 2017;5:23.
34. Xie H, Cheng Y, Chen Y, Qian M, Xia Y, Chen C. Improvement in the bonding of Y-TZP by room-temperature ultrasonic HF etching. *J Adhes Dent.* 2017;19:425-33.
35. Sriamporn T, Thamrongananskul N, Busabok C, Poolthong S, Uo M, Tagami J. Dental zirconia can be etched by hydrofluoric acid. *Dent Mater J.* 2014;33:79-85.
36. Lee H, Woo C, Paek J, Geis-Gerstorfer J. Effects of acid treatment with ultrasonic on resin-zirconia bond strength. 40th Eur Prosthodont Assoc 65th Ger Soc Prosthet Dent Biomater. 2016. Available at: [http://www.rhsupplies.com/wp-content/uploads/2018/06/TestReport\\_Research-literature-Germany.pdf](http://www.rhsupplies.com/wp-content/uploads/2018/06/TestReport_Research-literature-Germany.pdf)

37. Zandparsa R, Talua NA, Finkelman MD, Schaus SE. An In Vitro comparison of shear bond strength of zirconia to enamel using different surface treatments. *J Prosthodont.* 2014;23:117-23.
38. Xie H, Chen C, Dai W, Chen G, Zhang F. In vitro short-term bonding performance of zirconia treated with hot acid etching and primer conditioning. *Dent Mater J.* 2013;32:928-38.
39. Hooshmand T, Van Noort R, Keshvad A. Bond durability of the resin-bonded and silane treated ceramic surface. *Dent Mater.* 2002;18:179-88.
40. Aung SSMP, Takagaki T, Ko AK, Halabi S, Sato T, Ikeda M, et al. Adhesion durability of dual-cure resin cements and acid-base resistant zone formation on human dentin. *Dent Mater.* 2019;35:945-52.

Alexandria Dental Journal