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Evaluation the effect of various surface treatment on the shear bond strength between zirconium and different composite materials

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Abstract

The clinical performance of zirconium restorations is directly related to the surface treatment and the properties and type of luting agent employed. The purpose of this study is to evaluate the effect of zirconia different surface treatments (primer, sandblast with 100µm Al_2O_3) on shear bond strength between zirconia surface and different composite materials (Z250 composite , Herculite composite, evereX composite). Seventy-two presintered zirconia cylinder specimens (GC, Austria) will be fabricated .The specimens were then randomly divided into three main groups (A) bonded with Herculite composite, (B) bonded with Z250 composite, (C) bonded with evereX composite each group has 24 then divided into nine subgroups. Bonding surface of zirconia cylinders were then luted with RelyX Ultimate adhesive resin cement. All specimens were subjected to shear loading force in a universal testing machine. The shear bond strength values were analyzed statistically with two-way ANOVA. When bonded the zirconium discs without surface treatment to the evereX composite material , the highest shear bond strength was obtained. The conclusion that using zirconium discs (GC) without surface treatment and bonded to the evereX composite material produced the highest shear bond strength followed by sandblasting the bonding surface of zirconium cylinders with 100µm Al_2O_3 and bonded to the Z250 composite material.

Keywords: Zirconium, Composite, Surface treatment, Shear bond.

Introduction

Recently, both patients and clinicians have been seeking superior esthetic metal free tooth colored restorations so the demand for metal free restoration had significantly increase (Cavalcanti, 2009). All ceramic restorations give the aesthetic pleasing restorations, obviously high effort has been made over the years to improve their brittleness and low tensile strength. Zirconium oxide–based materials such as yttria-tetragonal zirconia polycrystals (Y-TZP) were introduced for prosthetic rehabilitations as a core material for single crowns, conventional and resin bonded fixed partial dentures (Kansu and Aydin, 1996) and, in dental implantology , as abutments or implants and the combination of Y-TZP and computer-aided design/ computer-aided manufacture (CAD/CAM) systems reduces the number of steps in prosthetic manufacturing and eliminates the variables introduced by the manual procedures of the dental technician (Blatz, 2002). Y-TZP exhibits exceptional physical and mechanical properties, such as high flexural strength, fracture toughness, hardness, wear and corrosion resistance in acidic and basic ambient conditions, translucency, colour stability, greater effectiveness of diagnostic radiographs and high biocompatibility (Odman and Andersson 2001; Raigrodski , 2004; Pjetursson *et al.,* 2007). Moreover, the polycrystalline structure, which lacks a glass matrix, makes zirconia ceramic more resistant to hydrofluoridric acid etching and, as a consequence, resistant to chemical roughening (Guazzato *et al.,* 2005).

However, it was difficult to achieve a good bonding with zirconium. There are several reasons which can explain the weak bonding including mismatch of the coefficient of thermal expansion between the veneering ceramic and the zirconia substructure, porosity of the zirconia surface, improper support of the zirconia substructure, and residual stress from cooling (Ozcan, 2002). Different approaches have been used to enhance the bond between the zirconia and resin cements, such as coating methods (Zawta, 2001), a selective infiltration-etching technique (Toksavul, 2004). Phosphate ester monomer, 10-methacryloyloxydecyl dihydrogen phosphate

(MDP) based materials, surface roughening by airborne-particle abrasion, and surface roughening by the use of laser (Raigrodski, 2005).

As the popularity of all-ceramic restoration has been increased, a wide variety of resin cements has been developed in order to provide optimal bond strength and better esthetics (Nagayassu *et al.*, 2006).

Materials and Methods

Fabrication of zirconium samples: A total of seventy-two zirconium cylinders were made from presintered cold Isostatic Pressed zirconium discs (GC, Austria) with dimension of (5.5mm width, 3mm thickness) Figure (1), by cutting the discs into many rectangles, each rectangle was glued into the fitting pin the fitting pin was then introduce into the designated place in the milling–machine. A straight hand piece with a diamond disc operating at high speed was fixed to the movable member of the milling machine in a way allowing back and forth free movement of it along the side of blank of zirconium, in this way each blank was cut into a cylinder shaped blank of a diameter of 6.8mm in diameter.

Figure(1): GC zirconium disc.

Each cylindrical blank was cut by a diamond cutting disk into 5 cylinders. The cylinders were then sintered in tube furnace (ceramill therm, Aman Girbach)) at 1450 ^{0}C for 9.5 hours including cooling, according to manufacturer's instructions. During this process a 3-dimensional volumetric shrinkage of the milled cylinder of approximately 10% took place that is why the cylinders were milled approximately 10% larger. After sintering each zirconia cylinder was measured approximately (5.5mm in diameter, 3mm in height). The bonding surfaces of zirconia cylinders were then ground flat using a grinding machine (made in Iraq) and polished consecutively with 100, 200, 400, 600, 800 and1000-grit silicon carbide abrasive papers (Al-Alamain Ghalib, KSA) under water cooling to obtain standardized surface roughness (Akın *et al.,* 2011), to facilitate handling the zirconia cylinder during the polishing process a custom made holder was fabricated. The zirconium discs were then cleaned in ultrasonic bath (CODY, CD-2800, China) containing distilled water for 5 minutes, and then air dried (Patel *et al.*, 2011). Surface roughness for each samples was then confirmed by the use of the profilometer (Mahr, USA) to ensure standardization. A specially designed square rubber mold (made in Iraq) was used for the construction of the acrylic blocks. The mold was with dimensions of (1.5cm x 1.5cm x 1.5cm) and has a circular hole in the center of its base with a diameter of 7.5mm and a depth of 3mm. After mixing the monomer (Ivoclar Vivadent, Liechtenstein) and polymer (Ivoclar Vivadent, Liechtenstein), it poured in the mold and to make three different groups of acrylic blocks , the monomer mixing with oil pigment to get different colors of acrylic blocks. A seventy–two acrylic blocks where obtained, then divided into three equal groups . The yellow twenty-four acrylic blocks filled with evereX posterior composite material (GC, Japan), the blue group filled with Herculite® Ultra™ Composite material (Kerr, Italy) by placed 2mm of material then filled the rest of cavity and the last pink group filled with Filtek Z250 XT Universal Restorative Material (3M ESPE, Germany) by applied 2.5mm of material then filled the rest of cavity and all these procedure made following manufacturing instruction and cured for 20 sec for each increment.

Grouping of the samples: The zirconium discs were randomly divided into three groups depending on the surface treatment that would be used . Each group contained twenty-four discs. The zirconium discs then bonded to the large discs made from different composite material using scotchbond (3M ESPE, Germany) and RelyX ultimate cements(3M ESPE, Germany).

Group A_1: (n = 8) Zirconium discs were left without surface treatment bonded on top of the Herculite® Ultra™ Composite material(Kerr, Italy) discs surface using multipurpose scotchbond (3M ESPE, Germany) and RelyX Ultimate cements(3M ESPE, Germany). under a fixed load of 750g(Iraqi made) . Excess cement was wiped off using microbrush (made in China).

Group A_2:(n=8) The same as group A_1 but the zirconium discs were bonded on top of the surface of Filtek Z250 XT Universal Restorative Material disc(3M ESPE, Germany).

Group A_3: (n=8) The same procedure as group A_2 but the zirconium discs were bonded on top of the surface of everеX GC composite material discs(GC, Japan).

Group B_1:(n=8) The surface of zirconia discs were sandblasted for 15sec with 100μm aluminum oxide particles (Cobra, Renfert- GmbH., Germany) at a distance of 10mm between the surface of the zirconia cylinder and the blasting tip of the airborne-particle hand-piece and at 2.5 bars pressure prior to bonding procedures then bonded to the Herculite® Ultra™ Composite material discs(Kerr, Italy).

Group B_2: (n=8) Same procedure as that for Group B_1 was followed but the surface of zirconia discs were bonded to the Filtek Z250 XT Universal Restorative Material disc (3M ESPE, Germany).

Group B_3:(n=8) Same procedure as that for Group B_2 but the zirconia disc was bonded to the of everеX (GC, Japan) composite material discs.

Group C_1: (n=8) Same procedure as that for Group B_1 but the sandblasted zirconia disc was coated with adhesive agent (Multilink primers A and B, Ivoclar vivadent, , Liechtenstein) for 20 sec by using microbrush prior to bonding procedures.

Group C_2: (n=8) Same procedure as that for Group B_2 but the sandblasted zirconia disc was coated with adhesive agent (Multilink primers A and B, Ivoclar vivadent, , Liechtenstein) for 20 sec by using microbrush prior to bonding procedures.

Group C_3: (n=8) Same procedure as that for Group B_3 but the sandblasted zirconia disc was coated with adhesive agent (Multilink primers A and B, Ivoclar vivadent, Liechtenstein) for 20sec by using microbrush prior to bonding procedures.

Treatment of the bonding surface of the zirconia cylinder:

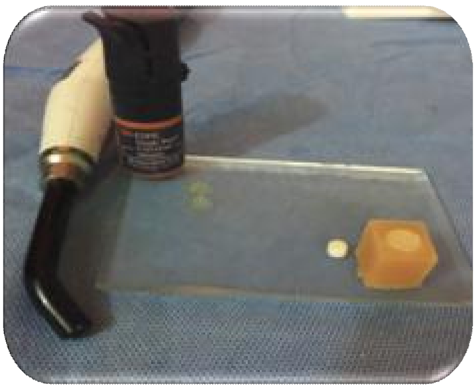
A- Sandblast treatment: Zirconia cylinders were mounted in a special holder so that the blasting tip was in a straight line with the sample at a distance of 10mm from the surface of the zirconia cylinder. The surfaces of the specimens were air particle abraded for 15 sec with 100μm Al_2O_3 particles(Cobra, Renfert- GmbH., Germany) at 2.5 bars (Re *et al.,* 2008) Then, the specimens were cleaned with 70% ethanol by wiping their surfaces with cotton and subsequently cleaning them for five minutes in an ultrasonic bath (CODY, CD-2800, China) with ethanol (Fischer *et al*., 2008) (Figure 2).

Figure(2): Placing the blasting tip perpendicular on the zirconium discs.

B- Application of primer: A saline containing primer (A+B, Ivoclar Vivadent, Liechtenstein) was applied by microbrush on the bonding surface area of zirconium discs, and allowed to react for 20sec and sprayed by a gentle steam of air (Figure 3).

Figure(3): Multilink Automix primer(A+B).

Cementation procedure: In order to standardize the cementation procedure, an adhesive tape (made in China) has a hole of 5.5mm in diameter was applied onto the exposed surface of the large discs prior to cementation. The hole was made using a paper piercer(made in China). This adhesive tape was used to restrict the area of bonding and help in removing the excess of cement (Patel *et al.,* 2011). The (Single Bond Universal Adhesive, 3M ESPE) was applied by microbrush on the surface of zirconium discs and different composite discs and allowed to cured for 20 sec then sprayed by a gentle steam of air, then RelyX Ultimate cements (3M ESPE, Germany) were placed according to the manufacturer's directions on paper pads and by using mixing spatula they were mixed and applied to the exposed surface of the composite disc and cured for 20 sec from three different directions (Patel *et al.,* 2011) (Figure 4).



Figure(4): The zirconium and the composite discs bonded with scotchbond.

The bonding side of the zirconium disc from the same group was seated onto its respective area on the exposed surface of the large embedded disc, and a load of 750g was applied vertically on the zirconium disc for 5 min. with the aid of dental surveyor (Dentaurum, Germany) (Hummel and Kern, 2004).

A custom mold made from heavy body silicone was used to secure the samples to the horizontal table of the surveyor and the load was applied on the upper part of the vertical arm of the surveyor. This load was applied to avoid any internal gaps in the luting cement and to help in standardization of the cementation procedure (Dawood, 2014).

After cementation, all groups were stored in a dark water bath (made in Iraq) containing distilled water at 37°C for 24 hours and before tested (Marocho *et al*., 2012).

Shear bond strength test: The bonded samples were attached to a universal testing machine (Laryee WDW-50, China), Figure (5A), and subjected to a shear load using a stainless steel notched chisel at a crosshead speed of 0.1mm/min until failure occurred (Hara *et al.*, 2001).

The samples were placed in the lower jaw of the testing machine. The acrylic block was held in a horizontal position in such a way that the long axis of the `chisel was placed parallel to the exposed surface of the large disc. The end of the notched chisel was positioned at the interface between the two bonded discs, Figure (5B).

The specimen was tightened strongly at a 90 degree to the vertical plane during the application of the load. The load that caused failure was recorded for each specimen and shear bond strength was calculated by dividing the force at which the bond failure occurred by the specimen bonding area and expressed in MPa according to the following equation (Usman and Nisha, 2014):

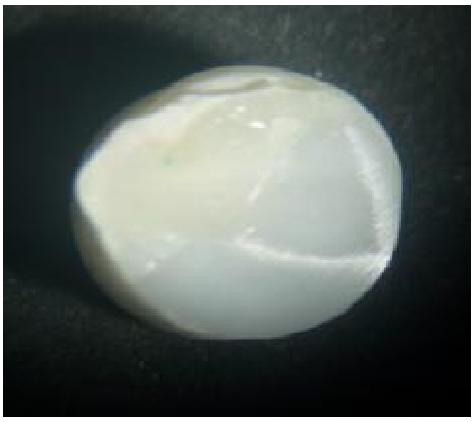
$$\text{shear bond strength (MPa)} = \text{Maximum force (N)} / \text{bonding area (mm}^2\text{)}.$$

(A) (B)

Figure(5): A: Universal Testing Machine, B: Load applied at the interface.

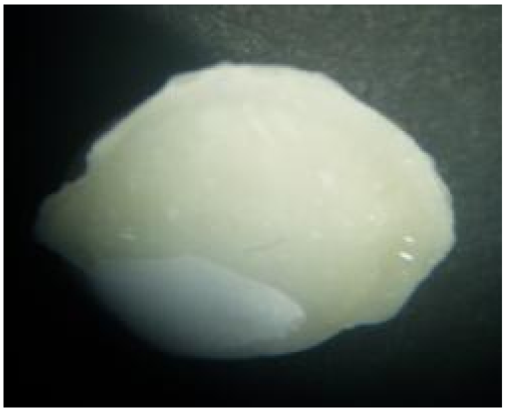
Evaluation Of The Mode Of Bond Failure: To determine the mode of failure after shear testing, the debonded surfaces were examined under a stereomicroscope (ST 60 series, China) at 20 X magnification. The failure modes were classified as follow (Nagayassu *et al.*, 2006; Dawood, 2014):

1- Adhesive failure: when all or most cement dislocate from the ceramic; more than 75% of the ceramic surface was visible as in Figure (6).



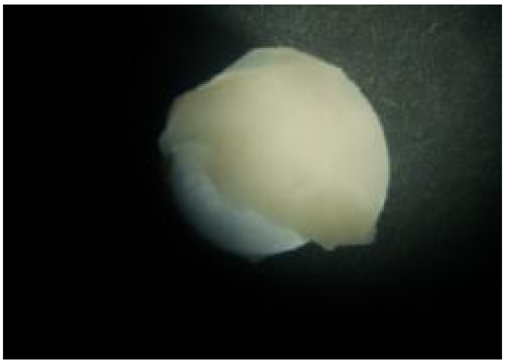
Figure(6): Adhesion failure.

2- Cohesive failure of the cement: When there was fracture in the cement layer with more than 75% of the ceramic surface covered with cement as in Figure (7).



Figure(7): Cohesion failure.

3- Cohesive failure of the composite: fractured ceramic adhered to the cement as in Figure (8).



Figure(8): Cohesion failure of composite.

4- Mixed Failure: When there was combination of adhesive and cohesive fractures as in Figure (9).

Figure(9): Mixed failure.

Statistical analysis: The data collected in this study were analyzed by using the program (SPSS for windows, version 20) and the following statistical measurements and tests were performed:

A- Descriptive statistics:
1- Mean.
2- Standard deviation.
3- Minimum and maximum values
4- Statistical tables.
5- Graphical presentation by Bar chart.

B- Inferential statistics:
1- Two-way ANOVA (analysis of variance) test was used to see whether there was any significant difference among the means of shear bond strength of the experimental groups.
2- Honest significant difference (Tukey HSD) test was performed to examine the source of difference between the groups in each state.

In the above tests , P values more than 0.05 considered as statistically non-significant, whereas P values less than or equal to 0.05 regarded as significant and P values less than 0.01 considered as statistically highly significant.

Results and Discussion

Descriptive statistics: The shear bond strength values for 72 samples from nine groups were measured and expressed in MPa for three different composite material and two different surface treatment. The means and standard deviations of shear bond strength values with minimum and maximum values for each group are shown in (Table 1).

Table (1): Descriptive statistics of shear bond strength (MPa) between the zirconium and the three composite materials (Z250, Herculite, evereX) discs using different zirconium surface treatment.

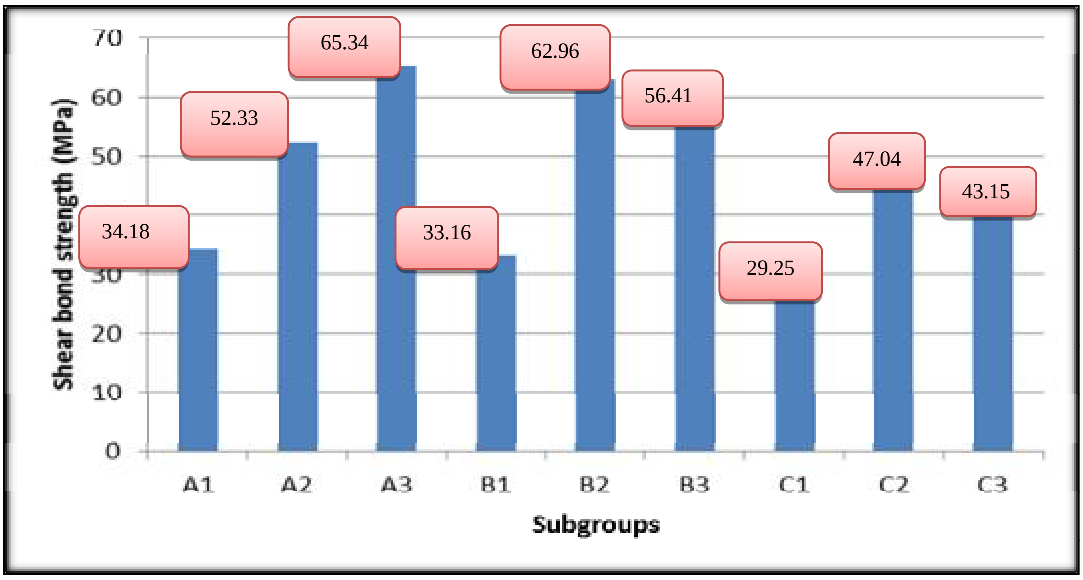
The type of composite and surface treatment	Groups	Subgroups	Descriptive statistics				
			N	Mean	±S.D	Min.	Max.
No treatment+ Herculite composite	A	A_1	8	34.18	3.78	29.8	38.9
No treatment + Z250 composite		A_2	8	52.33	3.23	49	58.9
No treatment + evereX composite		A_3	8	65.34	3.69	60	70.7
Sandblast + Herculite composite	B	B_1	8	33.16	2.43	30.1	37.4
Sandblast+ Z250 composite		B_2	8	62.96	2.46	60.5	68.3
Sandblast + evereX composite		B_3	8	56.41	4.63	50.9	61.9
Primer + Herculite composite	C	C_1	8	29.25	4.54	24.7	36.5
Primer + Z250 composite		C_2	8	47.04	2.85	43	50.9
Primer + evereX composite		C_3	8	43.15	3.49	40	48.7

These results showed that in group A, the highest mean of shear bond strength was in the subgroup A_3 with 65.34MPa (the minimum value 70.7MPa and the maximum value 60MPa) followed by A_2 with 52.33MPa(the minimum value 49MPa and the maximum value 58.9MPa) and the lowest mean found in the A_1 with 34.18 MPa (the minimum value 29.8MPa and the maximum value 38.9MPa), while in the group B the highest mean 62.96MPa in the subgroup B_2 (the minimum value 60.5MPa and the maximum value 68.3MPa) and the lowest mean was 33.16MPa in the subgroup B_1 (the minimum value 30.1 MPa and the maximum value 37.4MPa) while the mean in the subgroup B_3 was in between with 56.41MPa (the minimum value 50.9MPa and the maximum value 61.9MPa).

In group C the highest mean found in subgroup C_2 with 47.07MPa (the minimum value 43MPa and the maximum value 50.9MPa) followed by C_3 with 43.15MPa (the minimum value 40MPa and the maximum value 48.7MPa) and the lowest mean in C_1 with 29.25MPa (the minimum value 24.7MPa and the maximum value 36.5MPa).

Inferential statistics: The difference in the mean values for the subgroups (A, B, C) was tested by two way (ANOVA) test to see whether this difference was statistically significant or not (Table 2).

The results of this test showed that there is a statistically highly significant difference in the shear bond strength values for all the subgroups (A_1, A_2,A_3/B_1,B_2,B_3/ C_1,C_2,C_3). Further analysis by using the Honestly significant difference (HSD) test was performed for the subgroups to examine the source of difference (Table 3).



Figure(10): Bar-Chart of the mean values of shear bond strength.

Table (2): Comparison of the shear bond strength (MPa) among the subgroups

Groups	ANOVA	Sum of Squares	d.f.	Mean Square	F-test	p-value
A	Between Groups	3919.598	2	1959.799	153.458	0.000 (HS)
	Within Groups	268.189	21	12.771		
	Total	4187.786	23			
B	Between Groups	3924.013	2	19622.007	176.374	0.000 (HS)
	Within Groups	233.606	21	11.124		
	Total	4157.620	23			
C	Between Groups	1399.248	2	699.624	51.289	0.000 (HS)
	Within Groups	286.459	21	13.641		
	Total	1685.706	23			

Table(3): Tukey HSD test after ANOVA between subgroups

Groups	Subgroups		Mean difference	p-value
A	A_1	A_2	-18.15	0.000 (HS)
		A_3	-31.16	0.000 (HS)
	A_2	A_3	-13.01	0.000 (HS)
B	B_1	B_2	-29.80	0.000 (HS)
		B_3	-23.25	0.000 (HS)
	B_2	B_3	-6.55	0.002 (HS)
C	C_1	C_2	-17.79	0.000 (HS)
		C_3	-13.90	0.000 (HS)
	C_2	C_3	3.89	0.113 (NS)

The result showed that the differences between all the subgroups were highly significant statistically with exception that no significant difference between (C_2,C_3). To examine the effect of the surface treatment on the shear bond strength values in each composite materials, Tukey HSD test was applied (Table 4).

The result showed that Herculite composite group has the highest mean in the subgroup A_1 with 34.18MPa (the minimum value 29.8MPa and the maximum value 38.9MPa) followed by B_1 with 33.16MPa (the minimum value 30.1MPa and the maximum value 37.4MPa) and the lowest mean found in C_1 with 29.25MPa (the minimum value 29.8MPa and the maximum value 38.9MPa) .

In Z250 composite group, the highest mean found in B_2 with 62.96MPa (the minimum value 60.5MPa and the maximum value 68.3MPa) and the lowest mean found in C_2 with 47.07MPa (the minimum value 43MPa and the maximum value 50.9 MPa) but the subgroup A_2 was in between with 52.33MPa (the minimum value 49 MPa and the maximum value 58.9MPa).

In the last group that have evereX composite, the highest mean found in A_3 with 65.34 MPa (the minimum value 49MPa and the maximum value 58.9MPa) followed by B_3 with 56.41MPa (the minimum value 50.9MPa and the maximum value 61.9MPa) while C_3 the lowest mean with 43.15MPa (the minimum value 40MPa and the maximum value 48.7MPa).

The analysis by Tukey HSD test show that high significant difference between the different composite materials when applied sandblast surface treatment , no surface treatment applied or using primer.

The results of this test showed that there is a statistically significant difference in the shear bond strength values for the subgroups (A_1,A_2, A_3) and highly significant difference in the shear bond strength values for the subgroups (B_1, B_2, B_3, C_1, C_2, C_3).

Mode of failure: The mode of bond failure after shear bond test was observed under stereomicroscope and the results are summarized in (Table 7).

Table(4): The effect of different surface treatment on the shear bond strength of each composite material.

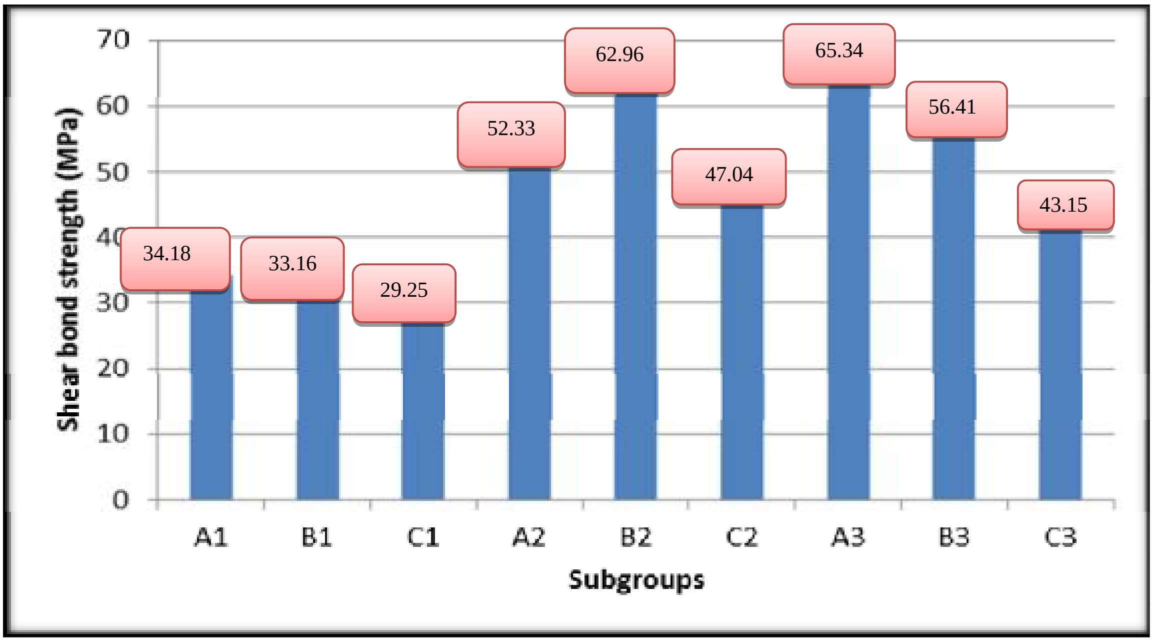
The type of composite	Subgroups	Descriptive statistics				
		N	Mean	+_ SD	Min.	Max.
Herculite composite	A_1:(No surface treatment)	8	34.18	3.78	29.8	38.9
	B_1:(Sandblast)	8	33.16	2.43	30.1	37.4
	C_1:(Primer)	8	29.25	4.54	24.7	36.5
Z250 composite	A_2:(No surface treatment)	8	52.33	3.23	49	58.9
	B_2:(Sandblast)	8	62.96	2.46	60.5	68.3
	C_2:(Primer)	8	47.04	2.85	43	50.9
evereX composite	A_3:(No surface treatment)	8	65.34	3.69	60	70.7
	B_3:(Sandblast)	8	56.41	4.63	50.9	61.9
	C_3:(Primer)	8	43.15	3.49	40	48.7

Table (5): Comparison of the shear bond strength (MPa) among the subgroups Tukey HSD test after ANOVA

Groups	Subgroups		Mean difference	p-value
A	A_1	A_2	-18.15	0.000 (HS)
		A_3	-31.16	0.000 (HS)
	A_2	A_3	-13.01	0.000 (HS)
B	B_1	B_2	-29.80	0.000 (HS)
		B3	-23.25	0.000 (HS)
	B_2	B_3	6.55	0.002 (HS)
C	C_1	C_2	-17.79	0.000 (HS)
		C_3	-13.90	0.000 (HS)
	C_2	C_3	3.89	0.113 (NS)

Table (6): Comparison of the shear bond strength (MPa) among the subgroups

Groups	ANOVA	Sum of Squares	d.f.	Mean Square	F- test	p-value
1	Between Groups	1052.586	2	526.293	63.992	0.000 (S)
	Within Groups	172.713	21	8.224		
	Total	1225.298	23			
2	Between Groups	108.236	2	54.118	3.987	0.034 (HS)
	Within Groups	285.074	21	13.575		
	Total	393.310	23			
3	Between Groups	1994.226	2	997.113	63.363	0.000 (HS)
	Within Groups	330.468	21	15.737		
	Total	2324.693	23			



Figure(11): Bar-chart the effect of different surface treatment on the shear bond strength (MPa) in each composite material.

Table (7): Modes of bond failure

Material	Subgroups	Adhesive failure (n)	Cohesive cement (n)	Cohesive zirconium (n)	Mixed failure (n)
Herculite composite	A_1	3	5	-----	-----
	B_1	2	5	-----	1
	C_1	2	6	-----	-----
Z250 composite	A_2	3	4	1	-----
	B_2	2	6	-----	-----
	C_2	4	3	1	-----
evereX composite	A_3	1	7	-----	-----
	B_3	3	4	-----	1
	C_3	2	5	1	-----

This table shows that the adhesive mode of failure was predominant in groups (C_2)which Z250 bonded to the zirconium discs treated with primer , however in the other groups, the cohesive failure in the cement was the predominant failure mode. The other types of bond failure occur less frequently.

Regardless of the zirconia microstructure, composition and surface texture is thought to play an important role in the resulting strength and clinical survival of the restoration. Several zirconia surface treatments have been introduced to improve the bond to the resin luting cement. Such as roughening the inner surfaces of zirconia restorations increases the available surface for the penetration of resin-based materials, improve the mechanical bond (Cavalcanti *et al.,* 2009). Even so the hydrofluoric acid etching and the application of a silane coupling agent to silica-based ceramics increases the bond strength between all-ceramic restorations and composite resins. These techniques do not increase the bond strength of zirconia and alumina ceramics because their high crystalline content them resistant to acid etching (Kosmac *et al.,* 1999).

Micromechanical interlocking and chemical bonding give strong resin bond to the zirconia surface (Blatz *et al.,* 2002). This is obtained by different surface treatment methods. Common options are grinding, airborne particle abrasion with aluminum oxide, acid etching, silica coating, and a combination of any of these methods. Initial zirconium disc (GC, Austria) is a yttrium-stabilized zirconia demonstrating a flexural strength of more than 900 MPa, and a fracture toughness of more than twice that of glass-ceramic materials. Suitable for restorations requiring high strength and exceptional durability. According to manufacturing instructions (Sturzenegger *et al.*, 2001).

The pre-sintered discs allow easy processing. Yet, once sintered to full density, its superior strength and inertness make it an ideal material for dental restorations. With these properties the discs meet the functional requirements requested by posterior masticatory forces (Lüthy *et al.,* 2005).

In the study used Hercultie XRV Ultradent composite (Kerr, Italy), it is indicated for anterior or posterior teeth and it has excellent handling properties and high wear resistance.

The other type of composite that used in this study was Filtek Z250(3M ESPE, Germany) that used for anterior and posterior restorations , core Build-ups and it has lowest polymerization shrinkage of leading composites, speed and convenience , excellent handling , outstanding Versatility.

The last type of composite used in this study was evereX posterior^{TM} (GC, Japan) indicated in high stress bearing areas such as molars and it has improvements in the load bearing capacity, the flexural strength and fracture toughness of dental composite resin reinforced with short E-glass fiber fillers in comparison with conventional particulate filler composite resin (Gahlert M. *et al.,* 2007).

The primer multilink A+B (Ivoclar vivadent, Liechtenstein) used in reinforced all-ceramics (zirconium and aluminium oxide) , produce high immediate bond strengths (Ivoclar vivadent, Liechtenstein).

RelyX Ultimate is new dual cured resin cement from 3M ESPE, as the manufacturers claimed it developed to meet the specific need for zirconium restorations. It is used Scotchbond universal adhesive which can be used in self-etch, selective etch or total etch approach. The Scotchbond universal can serve as adhesive to tooth substance as well as function as a saline , metal and zirconia primer there by simplifying clinical steps.

In this study the zirconium specimens were fabricated in disc like shape. This shape is selected instead of the rectangular one because it was found that when used the disc shape specimens, the stresses were distributed over the entire bond interface, while in the rectangular specimens the stresses will be concentrated at the corners and the central area (Phrukkanon *et al*., 1998).

In order to have a standard base line data for the surface roughness of all zirconia cylinders prior to treatment, The bonding surface of zirconia cylinders were then ground flat and polished using a grinding machine with water cooling to get standardized surface roughness (Akın *et al.,* 2011). Surface roughness for each samples were then confirmed by the use of the profilometer to guarantee surface roughness values to ensure standardization.

In this study sandblasting procedure was done with 100 µm Al*R*2*R*O*R*3,*R* as this particle size showed higher degree of surface roughness with less material removal from the internal surface that might interfere with internal detail of the restoration when comparing it when used other particle size. (Cavalcanti *et al.,* 2009), in addition larger particle size of Al*R*2*R*O*R*3*R* might create subcritical microcracks and transformation in the phases within zirconia surface which might negatively affect the mechanical properties (Ayad, *et al*., 2008; Karakoca and Yılmaz, 2009).

Zirconia cylinders were cleaned in ultrasonic device to ensure that the bonding surface was clean from any residue or contaminant prior to application of the primer and the luting agent. (Fischer *et al*., 2008).

Saline coupling agents were applied on the zirconium to mediate a chemical bond between the organic portion of the resin cement and the hydroxyl groups of the zirconium. The saline was applied as a single thin layer and then air dried (following the manufacturer's instructions) to remove the solvent and other byproducts. When saline applied as a single coat, it forms three different layers, but only the inner one is able to bond with the ceramic. The other two layers are hydrolysable and can adversely affect adhesion of the ceramic to the resin cement. So these two layers should be removed by a gentle steam of air (Murrillo and De Goes, 2014).

Significant strengthening of dental zirconium obtained when bonded to resin cement had been demonstrated by many in vitro studies (Pagniano *et al*., 2005; Fleming *et al*., 2006; Alakhras, 2011). So, the cementation process and bonding effectiveness play an essential role in the clinical success of all-ceramic restorations (Zareen *et al*., 2013).

Light-cured and dual-cured resin cements have been advocated for luting zirconium restorations. However, to ensure optimal bond strength, as well as optimal physical properties adequate polymerization of a resin luting agent is a crucial factor when cementing zirconium restoration (Hofmann *et al*., 2001). Incomplete polymerization of purely light cured resin luting cements has been demonstrated by previous studies due to attenuation of light energy by the restorative material (Braga *et al*., 2002; Akgungor *et al*., 2005). The type and thickness of the restorative material primarily affect the degree of the light attenuation and it has been advocated to use dual-cured resin luting cement for restorations thicker than 2mm (Akgungor *et al*., 2005). Thus, dual-cured resin cements were used in the present study to ensure adequate polymerization in areas that do not receive sufficient light.

According to several previous studies, a standard load of 750 g was applied onto zirconium specimens during the procedure of cementation to simulate the finger pressure applied clinically during cementation of zirconium restoration (Patel *et al*., 2011; Marocho *et al*., 2013; Passos *et al*., 2013).

Storing the bonded specimens in water at 37°C 24hrs used to investigate the durability of the bond strength by several previous studies (Wegner *et al*., 2002; Hooshmand *et al*., 2004; Salvio *et al*., 2007; Marocho *et al*., 2012).

There are different methods that can be used for evaluating the bond strength such as; 3-point bending test, tensile , micro-tensile test, the shear and micro-shear test (Blatz *et al*., 2003). The most commonly used method is the shear bond as it easy to perform, simple and can simulate shear stresses which are important contributors to bonding failure and deterioration of restorative materials (Blatz *et al*., 2010). Further advantage of the shear test is that it is performed without the need for sectioning procedures to obtain specimens, which may induce early micro-cracking (Scherrer *et al*., 2010).

The shear bond test different configurations are used to apply shear force like wire loops, notched chisel and knife-edge chisel. The test configuration is an important source of variation in the measurements of shear bond strength since it influences the stress distribution. In this study notched chisel was used since notched chisel and exert more even stress distribution at periphery of the bonding area as compared to knife-edge chisel which cause severe stress concentration at the load application area and result in recording lower bond strength (Salz and Bock, 2010). . In order to avoid cantilever effect on the adhesive surface during shear loading, the distance between the chisel edge and the adhesive surface is remain at 0.1mm during testing of all samples (Piwowarczyk *et al.,* 2011).

The test was conducted at a cross head speed of 1mm/min according to the ISO which recommend that in shear bond strength tests, the load should be applied with a cross-head speed of within 0.45 and 1.05 mm/min (ISO-TR 11405: 1994).

Table (1) showed that the highest values of shear bond strength obtained in the subgroup (A_3) when the zirconium discs that not undergo surface treatment bonding to evereX composite discs when comparing it to the other groups, that may be attributed to the fact that the pretreatment degraded the bond strength and this agree with Guazzato *et al*. (2005) and Uo *et al*. (2006) and the main composition of RelyX Ultimate phosphate is functional monomer. When phosphate monomer is applied to zirconia, the hydrogen group of phosphate monomer and the oxygen group of zirconia slowly react to produce water molecules and to form a stable Zr-O-P covalent bond and this conside with Yoshida *et al*. (2006) and Suh (2008) .The other fact that the fibers that found in the composition of evereX composite and their orientation should be considered as factors influencing the oxygen inhibition depth (Vallittu *et al*., 1997). The short-fiber composite used consisted of a cross-linked polymer network derived from dimethacrylate monomers (Bis-GMA and TEGDMA) and PMMA. During the polymerization process, this polymer matrix forms a semi-IPN structure (Vallittu *et al*., 2009). The differences between the short-fiber-reinforced composite and both dimethacrylate-based composites may be explained by the presence of fibers in short fiber-reinforced composite, which are shown to affect the oxygen inhibition depth and the internal void space formation . Orientation of the fibers favoured the passage of oxygen (Vallittu *et al*., 1997). The outcome for composite type was improved interlayer shear bond strength when the oxygen inhibition layer was present. This finding supports the influence of the physical surface properties of the oxygen inhibition layer on the bond strength (Koga K *et al.,* 2011; Oyama *et al*., 2012).

The second highest result showed in (B_2) when treating the bonding surface of zirconia with 100μm Al_2O_3 and bonded to Z250 composite material if comparing it to other groups , that may be attributed to the fact that treating zirconia bonding surface with sandblast increases surface roughness and undercuts. The result of this study agrees with Cavalcanti *et al.* (2009) who showed an increase in bond strength after air-abrasion with 100 μm Al_2O_3 and disagrees with De Oyague *et al.* (2009) Who concluded that air-abrasion on the bonding surface of zirconia did not produce higher bond strength, even though the substrate surface became rougher than the control group ,probably because of variety of grain size, or different pressure used in the study.

Studying the results of checking the bonding surface of zirconia, by using stereomicroscope at 20X magnification, table (7) greatly support the result of this study because it shows that mode of failures when there is no surface or using the sandblasting treatment was mostly cohesive failure and this creates high bonding to resin cement .

Conclusions

Within the limitation of this study, it was possible to conclude that:

1. Bonded evereX posterior (GC, Japan) composite to zirconium surface(GC, Asturia) without surface treatment produced highest shear bond strength.
2. Sandblasting of bonding surface of zirconia prior to cementation and bonded to Z250 (3M ESPE, Germany) composite material produced the higher shear bond strength values when comparing it when did not using surface treatment or applying primer.
3. Applying automix multilink primer (A+B) to the surface of zirconium discs before bonding to the evereX composite discs led to low value of the shear bond strength.
4. Bonded the zirconium (GC, Asturia) to evereX posterior (GC, Japan) give the high value of shear bond strength when treated the surface of the zirconium with sandblast or used primer even when no surface treatment applied.

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