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SHEAR BOND STRENGTH OF PRESSED E-MAX LAMINATE VENEER CEMENTED ON SURFACE TREATED ZIRCONIA SUBSTRATE

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ABSTRACT

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The purpose of this study was to evaluate the shear bond strength of E-max pressed laminate veneers discs cemented to the surface treated of zirconia core discs for repairing porcelain veneer chipping situations. A total of 40 Zirconia discs (n=40) were divided into four groups according to their surface treatment. Then 40 laminate veneer e-max press discs were constructed and subsequently cemented on the different treated surfaces. All specimen surfaces were prepared with a 30 μ m fine diamond rotary cutting instrument with water irrigation for 10 s and dried with oilfree air. Group 1: Control (n=10) where no surface treatment was applied. Group 2: The discs surfaces were treated using Cimara Zircon Repair system (Voco, Germany) (n=10). Group 3: The discs surfaces were conditioned with the zirconia primer Monobond plus (Ivoclar Vivadent) (n=10). Group 4: The disc surfaces were conditioned using CojetTM Repair system (3M ESPE) (n=10). Then each Laminate veneer was cemented in place using Rely x unicem self-adhesive universal resin cement following the manufacturer recommendations. The samples were stored for 24 hours in distilled water then subjected to shear bond strength test using a universal testing machine to measure the adhesion strength between bonded e-max and zirconia surfaces. The debonded surfaces were examined using SEMicroscope to reveal the failure nature for each group. The zirconia surfaces were analyzed using EDX (energy dispersive x-ray) to reveal the dominant elements left on the surface after debonding. Data were collected, tabulated and statistically analyzed with ANOVA test, followed by Tukey's post hoc pairwise comparisons (α =0.01). Zirconia surfaces treated with (Cimara Zircon Repair System) exhibited superior Shear bond strength values. With the limitation of this study it was found that all surface treatments used affected positively the bond strength between E-max laminate veneers and Zirconia.

KEY WORDS: Zirconia, Chipping repair, Zirconia primer, Shear bond strength, e-max press veneer, monobond plus, Cimara Zircon Repair system, Cojet Repair system, Rely-x unicem.

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All ceramic restorations are becoming part of many dental practices due to their superior esthetics and biocompatibility ⁽¹⁾. Metal substructures were widely replaced by high resistance porcelain core veneered using silica based porcelain. Among core materials used, Zirconia was the most predictable regarding mechanical properties ⁽²⁾. Unfortunately, due to the inherent differences in materials and behavior between veneering porcelain and their zirconia substrate 15% of cases reported chippings after 2 years' follow-up⁽³⁾. The exact reason of veneer chipping over zirconia is still unclear. Three factors generally contribute in the problem such as interfacial bonding, match of the core veneer materials and strength of the veneering ceramic. Also, the veneering technique has a crucial role in chipping due to the repeated cycles of firing in the oven ⁽⁴⁾. Delaminations with the exposure of core ceramics and minor chip-off fractures are cited as the most frequent reason for ceramic restoration failures⁽⁵⁾. Whereas numerous studies have reported failure modes of veneering porcelain fracture in allceramic crowns, only a limited number of studies have either presented or suggested solutions to post-clinical fractured status for existing situations ⁽⁶⁾. Several attempts were used to enhance bonding between composite resin cement and the zirconia surface such as mechanical grinding or sandblasting or chemical conditioning of the surface such as liners or silica coating to improve wettability and bonding ⁽⁷⁾. If the bond strength between these two materials was to be proven clinically acceptable, companies and clinicians would be able to avoid wasting the time, material, and money required to fabricate a new crown. A simple yet predictable way for porcelain chipping repair over zirconia represent a challenge for clinicians facing this problem. Dental product companies are developing types of composites for anterior region but due to their intrinsic nature; ceramics are more hydrophobic than composites and thus less prone to the influence

of various colorants and aging (8,9). Zirconia surface conditioning using liners with roughness by means of stones or sandblasting is claimed to be an effective way for bonding resins to zirconia ⁽¹⁰⁾. Porcelain veneers are well known to be superior over composite veneers in durability, strength and excellent biocompatibility. The translucent properties of porcelain allow it to mimic the light handling characteristics of enamel giving it a sense of depth which is not possible with other bonded esthetic materials such as composites (11). Porcelain veneers are also smooth thus resisting permanent staining due to smoking and coffee consumption. Pressed lithium dissilicate laminate veneers proved to possess superior mechanical properties as well as excellent fit and esthetics (12). With the steady evolution in bonding to zirconia substrate using liners, bonding ceramic laminates to zirconia will become soon a reality. Due to different bond strengths depending on the exposed surface in the fracture $^{(13)}$, it is valuable to investigate the bond strength of the porcelain-resin repair system onto Zirconia cores using several surface treatments. With the clinicians repeated need to repair moderate to large porcelain chippings over zirconia by means of a simple and reliable way, this study was intended to enlighten the feasibility of e-max porcelain laminate bonding to zirconia evaluating that such a porcelain repair system can serve as an efficient, cost-effective, and conservative solution when applied .

MATERIALS AND METHODS

Preparation of Zirconia discs:

A total of 40 Zirconia discs 10 mm diameter and 3mm thickness were constructed using Cad-Cam system (Laserdenta Gmbh Germany). A copper mold with an inner diameter of 10 mm was scanned using (openscanstripe) Fig (1). Then discs were designed to fit in the mold using exocad's CAD software Fig (2, 3). After milling the samples using prefabricated blocks of zirconia (Prettau Zirkon; Zirkonzhan, Italy) were sintered using a fast sintering furnace (Denta-star s1 plus). All the samples were embedded in the center of cold cure acrylic resin molds (Acrostone dental factory) in a stainless-steel holder leaving the repair surfaces uncovered. Finally, the samples were finished according to the manufacturer recommendations using turbines.

Clinically, when the fracture site is repaired with

Composite resin, surface grinding with a diamond bur is a mostly used for surface cleaning to improve mechanical bonding and remove the contamination layer as a standard procedure to simulate this clinical condition, all specimen surfaces were prepared with a 30 μ m fine diamond rotary cutting instrument (komet, Gmbh) with water irrigation for 10 s and dried with oil-free air.

All discs were ultrasonically cleaned in 96% isopropyl alcohol for 3 min and steam-cleaned for 10 s then randomly grouped into 4 groups according to their surface conditioning:

Table I describing groups and procedures done:

Group1: Control (n=10) Served as a control group and no surface treatment was applied.

Group2: The discs surfaces were treated using Cimara Zircon Repair system (Voco, Germany) (n=10).

Group3: The discs surfaces were conditioned with the zirconia primer Monobond plus (Ivoclar, Vivadent) (n=10)

Group4: The disc surfaces were conditioned using CojetTM Repair system (3M ESPE) (n=10). Each group treatment is described in table I.

Preparation of E-max press discs:

A total of 40 discs 8 mm diameter and 2mm thickness were constructed using E-max press. Each disc was pressed using E-max ingots color A2 after wax pattern was done in a copper mold having its inner diameter 8mm and 2 mm thickness Fig(4). Spruing and investing was done then the mold subsequently was burnt out and pressed by means of

Tested Groups	System name	Procedure		
		1.Surface grinding with a 30-mm fine diamond rotary cutting instrument with		
Group 1	Control	water irrigation for 10 s and dried with oil-free air		
		2.Application of Rely-x unicem and cementing of E-max press discs according to		
		manufacturer recommendations		
		1.Roughening the zirconia discs using the Cimara bur (10 strokes)		
	Cimara Zircon	2. Application of Cimara Primer then left for 60 seconds to react and air dry		
Group 2	Repair system	3.Application of Cimara Zircon Adhesive then light curing for 20 s		
	(Voco,Germany)	4. Application of Rely-x unicem and cementing of E-max press discs according to		
		manufacturer recommendations		
		1. A thin coat of Monobond Plus was applied with a micro-brush to the zirconia		
	Zirconia Primer	discs; the material was left to react for 60 seconds. The remaining was dispersed		
Group 3	Monobond Plus	with a strong stream of air.		
	Ivoclar Vivadent)	2. Application of Rely-x unicem and cementing of E-max press discs according to		
		manufacturer recommendations.		
		1. Sandblasted by silicate-coated alumina particles with a diameter of 30 mm at a		
Group 4		pressure of 2.3 bar and from a distance of 10 mm		
	Cojet Repair system	2. The tribochemical coating was completed by sialanisation using ESPESil for		
	(3M Espe)	(10 s)		
		3. Application of Rely-x unicem and cementing of E-max press discs according to		
		manufacturer recommendations.		

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Fig. (2)



Fig. (3)



a press porcelain furnace (Ney Ceram press Qex). Each disc was finished and glazed according to the manufacturer recommendations.

Samples cementation:

E-max discs were cleaned ultrasonically in 96% isopropyl alcohol for 3 min and then steamcleaned for 10 s. Each disc was surface treated by means of hydrofluoric acid 5% (Ivoclar, Vivadent GMBH) for 30 seconds then Silane coupling agent was applied using Micro-brushes (Monobond plus). The material was left to react for 60 seconds then the remaining was dispersed using a strong stream of air. The grouped Zirconia samples were prepared and cemented using Rely x Unicem self-adhesive universal resin cement, on a digital scale by finger pressure till the load reached 500 g. Excess cement was removed using micro-brushes and foam pellets. Finally, the samples were cured from all sides for 40 s at 1mm using a light polymerizing unit (Astralis 3 Ivoclar Vivadent) with an output power of 600 mW/ cm^{2.} All specimens were stored in distilled water at 37°C for 24 hours before testing.

SBS test:

The discs were then embedded in acrylic resin centralized within a special metallic attachment that was fixed to the lower compartment of the instron testing machine. The specimens were subjected to shear bond strength test using a universal testing machine (Instron 3345). A tapered wedge that was fixed to the upper compartment applied the load at a cross head speed of 1mm/min until debonding occurred (Fig: 5). Shear bond strength values were recorded in newtons and then converted to megapascals (MPa). The mean and standard deviation for each group were calculated from the obtained data.

Fractographic analysis:

After fracture, the debonded surfaces of each specimen was assessed using SEM Model Quanta 250 FEG Netherlands (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 K.V., magnification 14x up to 1000000 and resolution for Gun.1n). The failed surfaces were scanned using EDX to evaluate the remnants elements on the fractured surfaces and their percentage for the evaluated specimens.

Statistical analysis:

The results of the shear bond strength were

statistically analyzed. Data obtained from zirconia and the cemented e-max veneers were calculated using ANOVA test. Pairwise comparisons were made by Tukey post-hoc test. Analyses were carried out by the SPSS 15.0 (SPSS Inc., Chicago, IL, USA) with a p < 0.01 significance level.

RESULTS

Figure (6) represents the mean and standard deviation values of the shear bond strength test and the results of statistical analysis for all the groups. The results indicated that there were significant differences between all groups. The highest mean bond strength values were obtained in Group 2 (20.178 ± 2.3 MPa) while the lowest mean value was found in group I (9.879 ± 3.2 MPa). The difference between groups was statistically significant as indicated by ANOVA followed by pair-wise Tukey's post-hoc tests as shown in the tables (2-3) below:

TA	BI	Æ	(2)
			(-)

Groups	Count	Sum	Average	Variance
Control	10	98.79	9.879	0.675699
Cimara Zircon repair	10	201.78	20.178	1.440618
Monobond plus	10	148.97	14.897	0.667157
Cojet	10	199.7	19.97	2.175244

TABLE (3)

Treatments pair	Tukey HSD Q statistic	Tukey HSD p-value	Tukey HSD inferfence
Control vs Cimara zircon	29.2510	0.0010053	** p<0.01
Control vs Monobond plus	14.2520	0.0010053	** p<0.01
Control vs Cojet	28.6602	0.0010053	** p<0.01
Cimara vs Monobond plus	14.9990	0.0010053	** p<0.01
Cimara vs Cojet	0.5908	0.8999947	insignificant
Monobond plus vs Cojet	14.4082	0.0010053	** p<0.01



Fig. (5)

Interpretation of the SEM examination:

The SEM examination showed different mode of failures for the tested groups.

All samples showed mixed types of failures; adhesive and cohesive except the control group which represented an exclusive adhesive failure.



Cimara zircon repair group images showed a predominantly cohesive failure with small areas of adhesive failure.

The Cojet group images were obvious with an adhesive failure showing a rough topography emphasizing the particles deposited on the discs



Fig: (7) (A-D) SEM photos the debonded Zirconia surfaces of different surface treatments

examined surfaces with a thin layer of cement bonded to it.

Finally, the Monobond plus group images showed a mixed type of failure mainly cohesive with scattered islands of adhesive failure. All samples were represented in fig:7 (A-D).



Energy dispersive x-ray analysis:

Cimara bur EDX results

The inorganic part of different tested samples surfaces was investigated by means of EDX method. The investigation was performed for three points on each sample surface. All results were normalized to the unit. It was noticed during results examination that there were a lot of common chemical specimens with (some differences) as: carbon, oxygen, silica, Fluorine, Potassium and Barium. All samples have a higher percentage of oxygen (highest weight percentage value was obtained in the Cojet samples while the least values were obtained in Cimara bur samples): indicating the presence of copious amounts of oxides formed on the surface promoting bonding to zirconia samples. Some of the examined surfaces were showing the presence of heavy metals as Ba in the Cimara bur samples also some presence of Al and Si was obvious in the Cojet samples. Because of Alumina and Silicates content, bonding to Zirconia was improved giving higher shear bond strength values.

TABLE (4) Showing EDX results average values in wt.% for 3 pc	oints.
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Samples Chemical elements	Control	Cimara zircon repair	Monobond plus	Cojet repair system
С	28.26	28.66	25.63	24.29
0	21.89	17.76	18.13	24.02
F		3.77		2.75
Na				1.06
Al	2.4		1.73	3.42
Si		15.32	16.55	29.98
Р		1.01		1.62
Cl		0.48	0.96	
K		0.62		3.23
Ca		0.80	0.64	
Ba		7.87	4.56	3.23
Yb		19.45	6.44	6.40
Zr	47.45	4.28	25.36	

DISCUSSION

Adhesive bonding evolution in the recent years, gave rise to a new era of more conservative approach regarding repair of zirconia veneer chippings. Formerly, it was mandatory to replace the whole restoration leading to many financial, biologic and esthetic problems ⁽¹⁴⁾. The conventional repair with composite materials had many drawbacks, such as wear, surface polish longevity and more importantly shrinkage, which leaves the bonding interface subjected to detrimental stresses ⁽¹⁵⁾.

The idea of repairing zirconia with e-max press laminate veneers was to overcome the negative properties of composites previously mentioned. knowing that adhesion with silica based and glass ceramics gives a stable and reliable bond with methods conventionally used by means of ceramic etchants and silane coupling agents.^(16,17)

The main problem was to overcome the bonding weakness with zirconia surface and creating a durable and strong bond ⁽¹⁸⁾. Silane coupling agents applied to etched surfaces with hydrofluoric acid dissolving glass matrix of silica-based ceramics is inapplicable to zirconia ceramics as the etchant is not effective on its inert surface ^(19,20).

Sang JL et al⁽²¹⁾ demonstrated that the shear bond strength of composite to 50% surface of veneering ceramics was statistically higher than that of composite bonded to only core ceramics. Indicating that the more remaining veneer material the more the reliability of the bond to repair materials.

Pressed ceramics performed significantly better when comparing the shear bond strength of ceramics veneered onto zirconia core by pressing and layering technique⁽²²⁾.

Material surface roughness affect surface energy and wettability ^{(23),} surface treatments such as airborne particles or the use of stones with the addition of primers are used to increase bond strength of resin composites materials to all ceramics.

According to the results of the present study, The Cimara zircon repair system recorded the highest shear bond strength mean value (20.178± 2.3 MPa) followed by Cojet group with a mean value of (19.97± 3.2 MPa) while the Monobond plus group recorded the second lowest mean value with $(14.897 \pm 2.1 \text{ MPa})$. Finally, the least shear bond strength was obtained by the control group with a value of (9.879± 3.2 MPa). All groups showed statically significant difference except between Cimara zircon repair group and the Cojet group as shown in Table (3). The results of this study came in accordance with the previous one done by Uzun et al 2016 (24) showing that Cimara Zircon repair system showed the highest shear bond strength, with rougher surface.

Cimara zircon system resulted in a significantly rough surface on the zirconia substrates leading to much more wetting and larger surface area. The more reactive surface with the corresponding primer and adhesive led to significantly higher shear bond strength. The Cimara zircon system showed mostly cohesive failure within the cement explaining that the bond strength between zirconia and the luting cement was high enough to break the cement before debonding (25-26). The Edx results showed that the zirconia surfaces were loaded with different inorganic materials with different weight percentage showing amounts of carbon and oxygen giving more information regarding the reactive components on the surface. The primed zirconia surfaces showed different primer components as sodium (Na), aluminum (Al), silicon (Si), potassium (K), and oxygen (O), whereas in the uncoated specimen only zirconia (Zr) and oxygen were identified (27-28).

The Cojet system used in this study revealed the second highest shear bond strength. The zirconia substrate was blasted by silica coated alumina particles accelerated by compressed air, the Sio₂ penetrated and was embedded on the zirconia surface ⁽²⁵⁾. The embedded Silica particles increased

surface roughness and area along with the noticeable increase in the silica content as revealed by SEM and EDX analysis. By adding silane coupling agent a covalent bonding was achieved between the silica layer and the luting resin cement thus enhancing the bond strength ^{(29-30).} It was found also that MPS has shown enhanced bonding results in comparison with other silane coupling agents ⁽³¹⁾.

Monobond plus group showed an acceptable shear bond strength of $(14.897 \pm 2.1 \text{ MPa})$. The Primer was claimed to be a universal primer suitable for all dental alloys and ceramics. It has both a silane coupling agent and methacrylate monomers with functional phosphoric acid and sulfur compound groups as zirconium oxide has a high affinity to phosphoric acid. The bond recorded was adequate for intraoral repair in accordance with many previous researches ⁽³²⁻³³⁻³⁴⁾.

Rely x Unicem was chosen to be the luting cement for its outstanding clinical performance and ease of manipulation ⁽³⁵⁻³⁶⁾.

The results of the present study contradict the results obtained by Sharkawy A 2015⁽³⁷⁾ who found that Cimara grinding bur protocol was the worse giving the least bond strength among all tested repair systems. The author claimed these low values could be attributed to the lack of diamond abrasive particles on the Cimara grinding bur instead they are based on Silicium carbide that takes up the ground particles from the chipped ceramics giving less surface roughness.

The present study came in accordance with Kirmali et al ⁽³⁸⁾ concluding that the highest mean shear bond strength was obtained by the Cimara repair system with no significant difference with Cojet system repair this difference could be attributed to the different silane and bonding agents used for the different systems during their study.

This study found that recent surface conditioning techniques based on combination of micromechanical and chemical conditioning are effective in enhancing bond strength between composite and zirconia material ⁽³⁹⁻⁴⁰⁾. Further investigations are required to assess the longevity of the bond as well as the durability after thermocycling and fatigue.

CONCLUSIONS

Within the limitation of this study it was found that different methods used for zirconia surface treatments significantly increased the bond strength values of the veneering e-max press and could be considered as a reliable mean for repair after chipping. Further studies should be followed to investigate the stability and longevity of the bond in the oral environment.

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