



Influence of Different Self-Adhesive Resin Cements on Shear Bond Strength of Ceramic Orthodontic Bracket Bonded To Zirconia

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ABSTRACT

Objective:To evaluate the shear bond strength of self-adhesive cements containing different functional monomers when used to bond ceramic brackets to zirconia and to compare the effect of different surface pretreatments on shear bond strength. **Materials and Methods:**-A total of 48 Zirconia discs; each with a dimension of 8 x 8 x 3 mm were manufactured using CAD/CAM technology. The discs were randomly divided into 6 groups (n=8) as follows: Group C1: discs without pretreatment and primer application + Heliolit orthodontic composite, Group C2: discs with pretreatment with intraoral air abrasion and z-prime + Heliolit orthodontic composite, Group NC1: discs with no pretreatment and primer application + Multilink Speed resin cement, Group NC2: discs with no pretreatment and primer application + BeautiCem SA resin cement, Group PC1: discs with pretreatment with intraoral air abrasion and z-prime + Multilink Speed resin cement, Group PC2: discs with pretreatment with intraoral air abrasion and z-prime + BeautiCem SA resin cement. The ceramic brackets were bonded to the specimen and subjected to thermal cycling. Instron universal testing machine in which the mono-beveled chisel was directed vertically on the interface between the bracket base and specimen surface transmitting a compressive force with a cross head speed of 1mm/minute until the detachment of the bracket was used. The maximum failure load was recorded in Newton (N). The maximum failure load was then divided by bracket base surface area (10.55 mm²), to present the bond strength in MPa. Comparison of data between the groups was performed using two-way ANOVA followed by multiple t-tests for P values. **Results:** Multilink Speed and BeautiCEM SA resin cements, produced an adequate shear bond strength to zirconia without being excessive. (being over 13 MPa). Universal

adhesive with the pretreated zirconia and conventional orthodontic composite produced sufficient shear bond strength to withstand the orthodontic treatment.

Conclusions:Bonding ceramic brackets to zirconia substrate using resin cements, produced better shear bond strengths than using conventional orthodontic composite. Pretreatment of zirconia using sandblasting and a zirconia primer further enhanced the shear bond strength.

Keywords:Zirconia; Resin cements; ceramic brackets; surface modification.

I. INTRODUCTION

Orthodontic treatment helps restore the function and esthetics of teeth which has an effect on the face as a whole and on the quality of life of the patient as well. Thus, as dental awareness increased, the demand for adult orthodontics increased as well, so much that lots of people all over the world get their orthodontic treatment done when they are adults. ⁽¹⁾

Ceramic brackets which was introduced in the 1980s as a more esthetically pleasing alternative for metallic brackets. These ceramic brackets are almost exclusively composed of aluminum oxide (Alumina) present in 2 forms; One form is polycrystalline, made of sintered or fused aluminum oxide particles, and the other contains a single crystal of aluminum oxide (Monocrystalline). ⁽²⁾

Zirconium oxide (Zirconia) has become one of the most widely used ceramics as it combines both high strength and natural looking esthetics. In the past, it was strictly used as a core material for veneering porcelain but with the advancement in dental material technology, it is now used as a standalone material for the restoration of both anterior and posterior teeth. ⁽³⁻⁶⁾ Zirconia is a polycrystalline ceramic which cannot



be etched even by using hydrofluoric acid as it has no glassy matrix. This poses a challenge for orthodontists as because of that it does not easily provide proper bond strength for brackets especially ceramic ones that have lower bond strengths than their metallic counterparts. Reynolds⁽⁷⁾ stated that a minimum bond strength of 5.9–7.9 megapascals (MPa), could result in successful clinical bonding. This keeps the bracket attached to the tooth for the length of the treatment without being excessively strong as to not damage the underlying substrate while detaching the brackets when the treatment is finished.^(2,7-10)

An integral part of orthodontic treatment is the bracket adhesion, which needs to be reliable for a predictable result without suffering a loss of operator chair time, prolonged treatment time and a loss of money due to the need of replacing the brackets that fell off.^(5,11-15)

Different surface treatment modalities have been proposed for enhancing the bond strength of zirconia. The one which offered the best adhesive results was mechanochemical surface pretreatment which combined the use of sandblasting and chemical agents based on organophosphate monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), 6-methacryloyloxyhexyl phosphonoacetate (6-MHPA) or 4-methacryloyloxyethyl trimellitate anhydride (4-META) from which 10-MDP showed the most promising results.⁽¹⁶⁻²³⁾

Z-PRIME PLUS is a phosphate monomer and it contains a propriety formula of concentrated methacryloyloxydecyl dihydrogen phosphate (MDP) and carboxylic monomers formulated specific to zirconia, alumina, and metal. The versatility of these primers is claimed to be a compelling feature for use on many different indirect substrates⁽⁸⁾

Although various studies have been proposed to overcome bonding problems with zirconium, a consensus has not been reached yet, and data regarding the problem of bonding of orthodontic brackets to zirconia surface are still lacking.^(24- 32) For these reasons, the aim of this study was to compare the shear bond strength (SBS) of ceramic orthodontic brackets bonded to zirconia surfaces by using different self-adhesive resin cements and surface treatment. The null hypothesis of this study was that there was no difference in the shear bond strength of the orthodontic ceramic bracket bonded to zirconia surfaces using self-adhesive cements containing different functional monomers with different surface pretreatments.

II. MATERIALS AND METHODS

The approval of this study was obtained from the Mansoura university dental faculty ethics committee, Mansoura, Egypt. The approval number was (A04061222).

The materials used in this study is illustrated in Table (1).

Material	Company	Composition	Lot#
zirconium oxide IPS Emax ZirCAD	IvoclarVivadent AG, Liechtenstein	ZrO ₂ 87-95%, Y ₂ O ₃ 4-6%, HfO ₂ 1-5%, Al ₂ O ₃ 0.1-1%	Y38637
50 μm Aluminum Oxide powder	Zest Dental solutions, Carlsbad, California	50 μm Aluminum Oxide Powder AL ₂ O ₃	L12YD
Tetric N Bond Universal adhesive	IvoclarVivadent AG, Liechtenstein	Bisphenol A diglycidyl ether dimethacrylate, trimethylene glycol dimethacrylate	NEO5528
Z- primer Plus.	Bisco, Inc, Schaumburg, IL, USA.	Organophosphate and carboxylic acid, biphenyl dimethacrylate and hydroxyethyl methacrylate	B-6001P
luting composite resin for orthodontic brackets. (Heliosit Orthodontic).	IvoclarVivadent AG, Liechtenstein	The monomer matrix consists of urethane dimethacrylate, Bis-GMA and decandioldimethacrylate (85 wt%). The filler consists of highly dispersed silicon dioxide	185803518960



		(14 wt%). Additional contents are catalysts and stabilizers (1 wt%).	
Self-Adhesive resin cement (BeautiCem SA).	Shofu, Kyoto, Japan	2-Hydroxyethyl methacrylate Fluoro-alumino-silicate glass Zirconium silicate filler (amorphas) UDMA [Cas.No.72869-86-4] Carboxylic acid monomer, Phosphonate monomer Polymerization initiator	PN 3213
Self-Adhesive resin cement (Multilink Speed).	Ivoclar Vivadent AG, Liechtenstein	Dimethacrylates- Ytterbium trifluoride - Methacrylate monomer with phosphoric acid group, glass filler and silicon dioxide Initiators, stabilizers and pigments	114866249075.
Ceramic orthodontic brackets. (Symetri Clear™)	Ormco, <u>Orange, California, United States</u>	polycrystalline-alumina (PCA).	746-2206

Table (1) showing materials used in this study.

Methods:

Sample size calculation

Sample size was calculated by Power analysis and Sample size software (PASS software for Windows, Hintze, J. (2011). PASS 11. NCSS, LLC. Kaysville, Utah, USA)

Calculation relied upon a previous study by Naseh R. et al, ⁽¹¹⁾ which was similar to our work. Sample size was calculated at n=8 in each subgroup, considering $\alpha=0.05$, $\beta=0.2$, $S1=0.8$, $S2=0.5$ and study power=0.8, using sample size calculation formula.

Specimens Fabrication

-A total of 48 Zirconia discs; each with a dimension of 8 x 8 x 3 mm were manufactured using CAD/CAM technology.

All CAD/CAM blocks were glazed according to each manufacturer's recommendations to mimic the smooth surface that would be present when a crown is delivered intraorally and the non-glazed surface was marked for easier identification.⁽⁹⁾ (Figure 1, a and b).

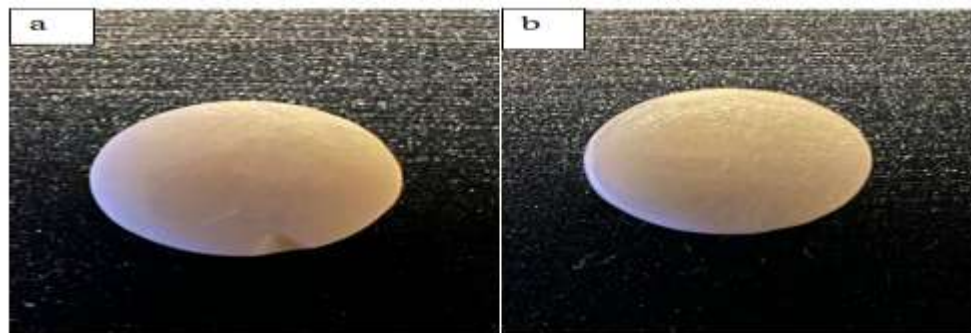


Figure (1): Showing the marked non-glazed (a) and the non-marked glazed sides of the zirconia discs (b).

(Figure2):

The discs were randomly divided into 6 groups (n=8) as follows,



1. Group C1: discs without pretreatment and primer application + Heliositorthodontic composite
2. Group C2: discs with pretreatment with intraoral air abrasion and z-prime + Heliositorthodontic composite
3. Group NC1: discs with no pretreatment and primer application+ Multilink Speed resin cement
4. Group NC2: discs with no pretreatment and primer application + BeautiCem SA resin cement
5. Group PC1: discs with pretreatment with intraoral air abrasion and z-prime + Multilink Speed resin cement
6. Group PC2: discs with pretreatment with intraoral air abrasion and z-prime + BeautiCem SA resin cement.

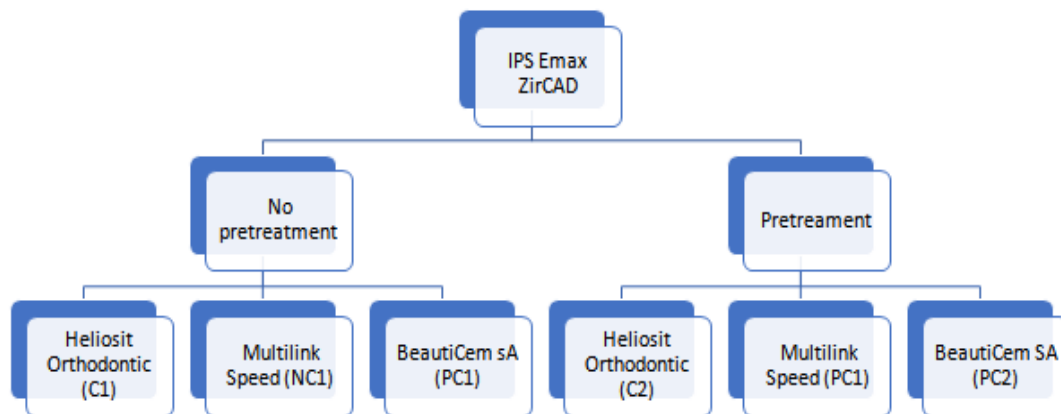


Figure (2): Showing the subgroups of the study (8 specimens each).

Surface treatment

Firstly, the surfaces of subgroups (C2, PC1, and PC2) were abraded with 50 μm aluminum oxide powder at a constant pressure of 2 bar for 15 seconds at 10 mm distance and 90-degree angle to the surface. The surfaces were then marked with a marker to ensure that the abrading powder reached the whole surface when the marking is removed.

Secondly, Z-prime was applied in a thin coat, and then it was air dried for 5 seconds to remove the solvent according to the manufacturer instructions.⁽¹³⁾

In groups (C1, NC1, and NC2) no surface treatment or primer application was done. While, in group C1, Tetric N Bond Universal was applied to the zirconia surface and to the bracket base. Then, ceramic central incisor orthodontic bracket was bonded to the specimen using Heliosit Orthodontic composite and light polymerized for 15 seconds at 1,100 mW/cm^2 (BlueLEX LD-107, Monitex industrial Co., Taipei, Taiwan).⁽¹⁹⁾

In group C2, Heliosit Orthodontic composite was used to bond the ceramic bracket after the previous surface conditioning according to the manufacturer instructions.⁽¹³⁾ On the other hand, in groups (NC1 and PC1) the same bonding procedure was done using Multilink Speed cement according to the manufacturer instructions.⁽¹⁵⁾ In groups (NC2 and PC2) the same bonding procedure

was done using BeautiCem SA cement according to the manufacturer instructions.⁽²³⁾

In groups (NC2 and PC2) the same bonding procedure was done using BeautiCem SA cement according to the manufacturer instructions.⁽²³⁾

All specimens were preserved and immersed in distilled water at 37°C for 3 months to mimic the clinical situation. (International organization for standardization- dental materials-Guidance on testing on adhesion to tooth structure⁽⁶⁷⁾ and werethermocycled for 3000 cycles in hot and cold baths at 5°-55°C \pm 4 °C for 30 seconds. Dual interval as a means of artificial aging was performed to simulate the oral environment prior to testing⁽⁷⁰⁾.

Specimens were then separately fixed on the lower fixed compartment of the Instron universal testing machine (Model 3345; Norwood, USA), in which the mono-beveled chisel was directed vertically on the interface between the bracket base and specimen surface transmitting a compressive force with a cross head speed of 1mm/minute until the detachment of the bracket. The maximum failure load was recorded in Newton (N). The maximum failure load was then divided by bracket base surface area (10.55 mm^2), which was measured using a digital caliper, to present the bond strength in MPa.⁽⁷²⁾



The SPSS statistical package for social science version 22 (SPSS Inc., Chicago, IL, USA) was used for data analysis. Shapiro Wilk was used to test the normal distribution of data. The data were parametric and met the normal distribution. Presentation of data was done using mean and standard deviation for statistical comparisons.

Comparison of data between the groups was performed using two-way ANOVA followed by multiple t-tests for P values. Bar charts were used for the graphical presentation of the data. $P < 0.05$ was significant.

III. RESULTS

Table (2): Comparison of SBS between C1 and C2.

Group	Mean \pm SD	Test of significance
		P value
C1	5.45 \pm 0.37	<0.001*
C2	11.45 \pm 0.28	

P value by t-test.

This table shows that:

C2 was significantly increased when compared to C1 [mean \pm SD 11.45 \pm 0.28 Vs 5.45 \pm 0.37 respectively; $P < 0.001$].

Table (3): Comparison of SBS between C1 and NC2.

Group	Mean \pm SD	Test of significance
		P value
C1	5.45 \pm 0.37	<0.001*
NC2	8.63 \pm 0.44	

P value by t-test.

This table shows that:

NC2 was significantly increased when compared to C1 [mean \pm SD 8.63 \pm 0.44 Vs 5.45 \pm 0.37 respectively; $P < 0.001$].

Table (4): Comparison of SBS between C2 and PC1 .

Group	Mean \pm SD	Test of significance
		P value
C2	11.45 \pm 0.28	<0.001*
PC1	13.25 \pm 0.48	

P value by t-test.

This table shows that:

PC1 was significantly increased when compared to C2 [mean \pm SD 13.25 \pm 0.48 Vs 11.45 \pm 0.28 respectively; $P < 0.001$].

Table (5): Comparison of SBS between C2 and PC2.

Group	Mean \pm SD	Test of significance
		P value
C2	11.45 \pm 0.28	<0.001*
PC2	13.4 \pm 0.42	



P value by t-test.

This table shows that:

PC2 was significantly increased when compared to C2 [mean±SD 13.4±0.42 Vs 11.45±0.28 respectively; P<0.001].

Table (6): Comparison of SBS between NC1 and PC1 .

Group	Mean±SD	Test of significance
		P value
NC1	8.76±0.32	<0.001*
PC1	13.25±0.48	

P value by t-test.

This table shows that:

PC1 was significantly increased when compared to NC1 [mean±SD 13.25±0.48 Vs 8.76±0.32 respectively; P<0.001].

Table (7): Comparison of SBS between NC2 and PC2 .

Group	Mean±SD	Test of significance
		P value
NC2	8.76±0.28	<0.001*
PC2	13.4±0.42	

P value by t-test.

This table shows that:

PC2 was significantly increased when compared to NC2 [mean±SD 13.4±0.42 Vs 8.76±0.28 respectively; P<0.001].

IV. DISCUSSION

In the realm of intermolecular bonding, diverse surfaces exhibit distinct levels of complexity. This is particularly true when sticking to atypical surfaces such as zirconia. Moreover, the process of affixing orthodontic brackets entails multiple intricate processes, rendering it a meticulous and time-intensive procedure. Hence, it became necessary to develop a more robust and expedient bonding procedure for attaching brackets to a zirconia substrate.

The objective of this study was to evaluate and compare the shear-bond strength (SBS) of several self-adhesive resin cements utilized for the bonding of ceramic brackets onto zirconia discs, in relation to the SBS exhibited by conventional orthodontic composite, commonly employed for orthodontic bracket luting purposes. The aforementioned process exhibited a higher degree of time consumption and susceptibility to debonding due to its involvement of a multitude of steps. Additionally, the purpose of this study was to investigate whether the application of pretreatment on the zirconia discs had any influence on the shear bond strength (SBS).

The efficacy of conventional orthodontic bracket bonding techniques is compromised when bonding to non-traditional surfaces like zirconia. This often leads to untimely bracket debonding, which hinders the progress of treatment, prolongs treatment duration, and consumes a significant amount of clinical chair time. Consequently, extensive research efforts have been undertaken to enhance the characteristics of dental materials and treatment methodologies, with the aim of establishing bracket bonds that exhibit enhanced stability and durability.^(70,71,72)

The results of this investigation revealed that both the self-adhesive resin cements yielded sufficient shear bond strengths for the purpose of bonding the bracket to a zirconia substrate. The shear bond strength (SBS) should possess a level of weakness that facilitates the convenient and safe removal of brackets, without causing harm to the underlying restoration.

Instead of using premolar brackets, which were not preferred, maxillary central incisor brackets were chosen for this investigation. The deviation from the flat surface of the ceramic discs by the premolar bracket's curvature has the potential to influence the SBS (shear bond strength)



values. The curvature of the central incisor brackets is relatively smaller, allowing them to conform to the flat surface of the ceramic discs.

UDMA based self-adhesive resin cements were found to be clinically successful when used to bond zirconia. Thus, it was needed to compare its efficacy in simplifying the procedure of bonding orthodontic brackets to zirconia crowns while still producing a sufficient bond strength for the orthodontic treatment.

A cementation device was used to standardize the forces placed during cementation as to eliminate the variable. After the cementation process, the samples underwent artificial ageing and thermocycling. The specimens underwent 3000 cycles of thermocycling. This was significantly higher compared to prior studies that assessed the bond strength between ceramics and brackets, which either did not subject the specimens to any thermocycling or performed a maximum of 500 cycles. A greater quantity of thermal cycles can more accurately represent the conditions of the oral environment and the decline in mechanical properties resulting from the ageing process.⁽⁵⁰⁻⁵⁵⁾

In the present study, the results showed that there was a significant difference between the SBS obtained using different luting agents and different surface treatment protocols.

According to our results, the control group (C1) did not produce adequate shear bond strength to bond brackets to zirconia as it produced SBS lower than 6 MPa. This can be owed to the fact that conventional orthodontic composite luting agent with a normal bonding protocol can not bond properly to zirconia surface as it is non-etchable and has a low reactive structure after sintering.

The findings of our study revealed that there was a highly significant difference between C1 and C2. C2 revealed greater shear bond strength than C1 which showed unacceptable SBS. The group (C2) had a higher SBS value which can be explained on the basis of the surface conditioning done to the zirconia by using Z prime which contains a combination of two active monomers, MDP, a phosphate monomer, and BPDPM, a carboxylate monomer that primes the surface of zirconia optimizing adhesive performance to zirconia.⁽⁷⁵⁻⁷⁶⁾

Moreover, the process of sandblasting the surface of zirconia leads to an elevation in surface energy, hence enhancing the adhesive properties of resin to zirconia. This observed improvement can be due to the increase in the roughness and hence the specific surface area, thereby decreasing the effective contact angle and increasing the wettability of luting material micromechanical

retention and enhancing the bond between hydroxyl groups of alumina and UDMA monomer of resin cement. This result is in agreement with a previous study done by Ji-Yeon Lee et al (80) and by Wolfart et al. (81)

Comparing NC1 and NC2 with C1 showed a highly significant difference between the groups. The observed phenomenon can be ascribed to the presence of an adhesive monomer comprising a long-chain methacrylate, accompanied by a phosphoric acid functional group. The presence of the phosphoric acid group facilitates the formation of a durable chemical connection with zirconium oxide.

Also, the result of this study confirmed that PC1 and PC2 are better than C2. This can be owed to the combination between the surface conditioning of the zirconia and the self-adhesive properties of the used luting agent.

Furthermore, our results showed a statistically significant difference between the groups PC1 and NC1 and PC2 and NC2 respectively. This showed that the surface conditioning of the zirconia with MDP primer (Z prime) and sandblasting have a monumental effect on the bonding procedure and confirms our previous findings.

V. CONCLUSIONS

- 1- Bonding ceramic brackets to zirconia substrate using resin cements, produced better shear bond strengths than using conventional orthodontic composite.
- 2- Pretreatment of zirconia using sandblasting and a zirconia primer further enhanced the shear bond strength.
- 3- Multilink Speed and BeautiCEM SA resin cements, produced an adequate shear bond strength to zirconia without being excessive. (being over 13 MPa).
- 4- Universal adhesive with the pretreated zirconia and conventional orthodontic composite produced sufficient shear bond strength to withstand the orthodontic treatment.

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