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Comparison of shear bond strength of metallic orthodontic brackets bonded to zirconia models underwent different surface conditioning methods and different primer systems

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ABSTRACT

Objectives: This study aimed to compare the shear bond strength (SBS) of metal brackets bonded to zirconia models with different surface treatment methods and two types of primers and to determine the adhesive remnant index (ARI). With the increase in demand for orthodontic treatment by adults and most adult patients having acrylic resin, amalgams, gold, composite resin, zirconia, or porcelain restorations, bonding of orthodontic braces to these surfaces is now a necessity. Brackets bonding to zirconia prostheses are a challenge in orthodontics because they need special surface conditioning.

Material and Methods: In this *in vitro* study, 60 zirconia models were divided randomly into two groups of 30 models according to the primer material used (Assure[®] Plus and Transbond[™] XT). The labial surface of each model was subjected to one of the following three surface preparation: No surface treatment (control group), sandblasting with 50 µm aluminum oxide (Al₂O₃) particles, and acid etching with 9.6% hydrofluoric acid (HF). Metal orthodontic brackets (Dentaurum) were bonded to zirconia models using Assure[®] Plus or Transbond[™] XT adhesives. The SBS was measured using a universal testing machine at a crosshead speed of o.5 mm/min. The labial surfaces of models were inspected under a stereomicroscope, and the ARI scores were determined. Raw data were analyzed using Statistical Package for the Social Sciences program through analysis of variance and the Kruskal–Wallis test (*P* ≤ 0.05).

Results: The Al_2O_3 air abrasion with the Assure[®] Plus group had the highest mean of SBS values, while HF groups with Transbond^{IM} XT adhesive or Assure[®] Plus gave rise a significantly lower SBS values than that obtained for the Al_2O_3 group. A significant difference was noted among the groups in the ARI scores. In Al_2O_3 group bonded with Transbond^{IM} XT had scores 1 and 2, which was designated as a mix-type failure, indicating a favorable failure mode.

Conclusion: This study showed that air abrasion of zirconia models had a significant effect on the SBS of metal brackets bonded to zirconia surface, and the Transbond[™] XT adhesive is a suitable primer material.

Keywords: Orthodontic metal bracket, Zirconia models, Surface conditioning method, Shear bond strength

INTRODUCTION

Direct bonding in orthodontics has decreased enamel decalcification, improved gingival health, and made the placement of orthodontic appliances more comfortable for orthodontists and

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patients.^[1] Nowadays, patients seek esthetic dentistry, so ceramic, zirconia, E.max crowns, or other types of fixed partial prosthesis meet their needs. With the increase in demand for orthodontic treatment for adult patients, orthodontists are often challenged with the problem of bonding orthodontic brackets to different types of prosthesis.^[2] Based on this evidence, numerous methods to improve bracket bonding to such restoration have been suggested, like mechanical (diamond bur, abrasive discs, air-particle abrasion, or laser), chemical (orthophosphoric acid, hydrofluoric acid [HF], maleic acid, or silane), or combinations of both methods to alter the surface characteristics of porcelain to withstanding orthodontic forces and provide sufficient bond strength.^[3,4] HF acid etching is typically utilized to improve the bracket bonding to traditional ceramics.^[5] Quentin et al.^[6] concluded that 40% HF is the most appropriate concentration for conditioning zirconia at ambient temperature because it forms the fastest and most uniform etching. Air particle abrasion is a technique in which aluminum oxide (Al₂O₃) particles, generally 50 µm, are projected to create abrasion by high air pressure on the surface of ceramic or another fixed prosthesis.^[7] During brackets bonding, the use of primer is highly recommended; many commercial porcelain or zirconia primers are available that are used to treat glazed surfaces and provide a strong bond by increasing the wettability of the ceramic or zirconia surface for bonding of adhesive material.^[8] The manufacturer claims that Assure® Plus is a recently introduced universal adhesive with high bond strength to normal enamel as well as to irregular metal surfaces such as gold, amalgam, stainless steel, ceramic, zirconia, and e.max pontics. Assure® Plus can be polymerized by chemical curing, light-curing, and dual-curing systems.^[9-11] As already said, providing reliable bonding between the bracket and the surface of restoration is necessary. This connection should be strong enough to prevent bonding failure by orthodontic force or by masticatory force and to protect the integrity of restoration during the deboning of brackets at the end of orthodontic treatment.^[12] The objective of this study was to compare the shear bond strength (SBS) of metal brackets bonded to glazed zirconia models using the two surface conditioning methods and two different primer materials.

MATERIAL AND METHODS

The ethical approval with Ref. no. (UoM.Dent.23/33) for this research was obtained from the Research Ethics Committee of the College of Dentistry/Mosul University.

The sample

The investigated sample included 60 Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) glazed zirconia models; each model consisted of two parts [Figure 1]; the upper part of the model is a crown of upper

left central incisor with a diameter determined according to Ash and Nelson^[13] and Sangalli *et al.*^[14] The cylindrical base with a diameter of 10 mm in height and a radius length of 10 mm. The sample size was calculated using sample size calculation formulas by Charan *et al.*^[15] and based on a study done by Mehta *et al.*^[16]

Zirconia models fabrication

A three-dimensional program (Exocad Galawy) was used to design the samples. Subsequently, the zirconia models were milled using the Go2dent digital software (Go2dental program) and a CAD/CAM milling machine (Maxx200, Korea). All the steps of laboratory processes for the model's design, construction, and glazing are carried out according to the manufacturer's recommendations by a single dental technician to ensure consistency. All the models were cleaned with a polishing paste without fluoride and then thoroughly washed and dried by the air for 5 seconds.

Criteria of sample selection

The labial surface of models was examined by a stereomicroscope (Japan/Union/ME3138) under ×10 magnification power [Figure 2] to confirm that selected



Figure 1: Zirconia models.



Figure 2: Evaluation of zirconia models by stereomicroscope.

models were clear of any impurities, porosities, cracks, or irregularities.^[17]

Sample grouping

The models were randomly divided into two main groups according to adhesive type: Assure[®] Plus bonding group and TransbondTM XT adhesive group. Then, each group was subdivided into three subgroups according to surface treatment methods: follow control group, HF treatment group, and Al₂O₃ air abrasion group.

Surface treatment procedure

For HF groups, the middle third of the labial surface of the models was treated with 9.6% HF acid for 1 min, then rinsed for 30 s and air-dried.^[18]

For Al_2O_3 groups: The models are fixed in a special design base to ensure standardization of distance and direction between microetcher (Ortho Technology, Emergo Europa) and model surface, as shown in [Figure 3]. Then, the middle third of the labial surface of models underwent air abrasion with 50 μ m Al_2O_3 particles using the microetcher at a distance of 10 mm and in a direction perpendicular to the labial surface of specimens with the pressure of 0.25 Mps for 15 s. Then, the models were rinsed thoroughly under tap water to remove Al_2O_3 particles and then air dried.^[19]

Bonding the brackets

For the groups of Transbond[™] XT orthodontic primer, a thin layer of adhesive primer (Transbond XT; 3 M Unitek[®], Monrovia, CA, USA) was applied and well-distributed on the center of the middle third of the labial surface of the crown of the model and left for 30 s and then dry with oil-free air for 10 s to remove excess Then, curing started using LED light curing device for 10 s according to manufactured instruction.

For the Assure[®] Plus groups, a single coat of the Assure[®] Plus primer was applied and well distributed on the center of the middle third of the labial surface of the crown of the model and left for 2 min, then thoroughly dry for 3–5 s and then curing started using LED light curing device for 10 s. All these procedures followed the manufactured instructions.

In all groups, the adhesive paste Transbond[™] XT (Transbond XT; 3 M Unitek[®], Monrovia, CA, USA) was applied over the base of the upper central incisor bracket (standard Edgewise 0.022 inch slot metal bracket, Dentaurum). Subsequently, the brackets were positioned at the treated center of the labial surface of the crown of the model at a distance of 4 from the incisor edge. Boons gauge was used to ensure the correct bracket position [Figure 4]. After that, the model is insulted on a customized mold and transferred to the stage of the universal testing machine. A universal testing machine applied a load of 200 g at a bracket slot for 10 s to confirm uniform adhesive thickness^[20] [Figure 5]. A sharp dental explorer removed the excess resin. Then, the adhesive



Figure 4: Bracket positioning.



Figure 3: Air abrasion of models.



Figure 5: A stable pressure is applied to the bracket.

was photopolymerized using an LED light curing device with a wavelength of (420–480 nm) and an illumination of (1200–1500) mw/cm². The curing light was applied for 20 seconds for the mesial side and 20 seconds for the distal side; the tip of the curing device was at a distance of 2mm



Figure 6: Shear bonding strength measurement by universal testing machine.



Figure 7: Adhesive remnant index score 3.

from the mesial and distal edges of the bracket base.^[21] The specimens were allowed to bench rest for 30 min and then placed in a sealed container containing distilled water and stored in an incubator at 37°C for 24 h before testing.^[22]

SBS measurement

The SBS test was measured using the universal testing machine (GESTER, Fujian, China) at the postgraduate laboratory, College of Dentistry, University of Mosul, with a crosshead speed of 0.5 mm/min. A prefabricated holder for the specimens has been constructed to ensure proper and secure seating of the specimen so that the bracket base is parallel to the direction of the shear force [Figure 6]. The chisel-shaped blade was directed toward the tooth--bracket interface in an occlusal-gingival direction. The necessary load to debond or initiate bracket failure was recorded in the Newton unit and converted to the MPa unit by dividing the failure load or force in the Newton unit by the surface area of the bonded bracket base (mm²).

Adhesive remnant index (ARI) measurement

After debonding of the brackets, the labial surface of the crown of the models was examined under Stereomicroscope at $\times 10$ magnification power (Optica, Italy) to assess the amount of adhesive material left on the model's surfaces. The criteria that were used for measuring ARI scores were as follows:^[10]

- Score 0 = No adhesive score remnant on the labial surface of the model.
- Score 1 = Less than half of the adhesive remained on the labial surface of the model.
- Score 2 = More than half of the adhesive remained on the labial surface of the model.
- Score 3 = All of the adhesive remained on the labial surface of the model, with a distinct impression of the bracket's mesh [Figure 7].

Statistical analysis

The Statistical Package for the Social Sciences Statistics V.19 software (New York, USA) was used to perform statistical

Table 1: Descriptive statistics and one-way analysis (ANOVA) of SBS (MPa) for all groups of zirconia models.									
	Primer type	Surface treatment method	n	Minimum	Maximum	Mean	Std. deviation	F	Sig.
Zirconia models	Assure [®] Plus	Control HF Al ₂ O ₃	10 10 10	2.78 3.90 17.02	3.70 4.30 17.98	3.3039 4.0989 17.6450	0.31672 0.14546 0.25779	1.037E4	0.000
	Transbond™ XT	Control HF Al ₂ O ₃	10 10 10	2.87 3.80 14.66	3.49 4.12 15.60	3.1865 4.0182 15.0527	0.22104 0.10698 0.29435	8.958E3	0.000
ANOVA: Analysis of Variance, SBS: Shear bond strength, HF: Hydrofluoric acid, Al ₂ O ₃ : Aluminum oxide, F:F value, Sig.: Statistical significance									

analyses. The Shapiro–Wilk test showed that the SBS raw data were normally distributed, and Levene's test confirmed homoscedasticity. One-way analysis of variance (ANOVA) was used to compare the mean SBS values of the groups at a significance level of $P \leq 0.05$, followed by Duncan's Multiple Range test. The non-parametric data of ARI scores were compared by the Kruskal–Wallis test.

RESULTS

Descriptive statistics and one-way analysis (ANOVA) of the SBS values of each group is shown in [Table 1]. The Al₂O₃ with Assure[®] Plus group had the highest mean value and revealed a significant difference between the means of SBS values of the groups at *P* ≤0.05. Using Duncan's Multiple Range test for SBS [Table 2], significant discrepancies were detected in Assure[®] Plus groups and Transbond[™] XT primer groups. The highest SBS was found in Al₂O₃ groups. The mean SBS for HF groups for Transbond[™] XT adhesive and Assure[®] Plus was lower than that one's obtained by Al₂O₃. Conversely, the control groups had the lowest mean SBS

Table 2: Duncan's multiple range tests for SBS (MPa).							
	Zirconia models						
	Assure [®] Plus	Transbond [™] XT					
Control							
Mean	3.3039°	3.1865 ^c					
п	10	10					
Std. deviation	0.31672	0.22104					
HF							
Mean	4.0989 ^b	4.0182 ^b					
п	10	10					
Std. deviation	0.14546	0.10698					
Al_2O_3							
Mean	17.6450 ^a	15.0527ª					
п	10	10					
Std. deviation	0.25779	0.29435					

SBS: Shear bond strength, HF: Hydrofluoric acid, Al₂O₃: Aluminum oxide, MPa: Megapascal, ^{a,b and} CDifferent letters vertically means statistical significant.

values [Table 2]. The independent *t*-test [Table 3] revealed that there was a significant difference at $P \le 0.05$ between Assure[®] Plus and TransbondTM XT primer type in Al₂O₃ groups, and there was no significant difference among other groups.

The distribution of the ARI scores among groups is illustrated in [Table 4]. The majority of the models in control and HF had scores of 0 and score 1 (all adhesive remained on the bracket base), while most of the samples of Assure[®] Plus with Al₂O₃ groups had scores of 2 and 3 (all the adhesive remained on the zirconia surface). The Kruskal–Wallis test revealed significant differences in the ARI scores among the groups at $P \le 0.05$, as shown in [Table 5].

DISCUSSION

Esthetic restorations such as zirconia crowns are highly requested for adults, new challenges are presented, like the bonding of orthodontic braces to zirconia surfaces. The ideal property of bonding material must be high enough to withstand the orthodontic forces during treatment and also allow debonding of the brackets at the end of treatment to maintain the integrity of the zirconia surface.^[3,23]

In the present study, when the zirconia models were roughened by 9.6% HF, the SBS was 4.0182 MPa for TransbondTM XT and 4.0989 MPa for Assure[®] Plus, which is lower than 5.9 and 7.8 MPa which is the reasonable clinical bond strength values of SBS that stated by Reynolds *et al.*^[24] The use of HF acid was investigated by several previous studies and reported that the application of HA on zirconia surface is not sufficient to provide adequate adhesion, as zirconia has a low silica content, which makes it resistant to acid etching and difficult to create porosities.^[1,25] According to Faria *et al.*,^[26] the HF provides no effect on the zirconia surface but provides adequate adhesive strength on glass ceramics, and this is due to differences in the composition of ceramics materials, which produce distinct topographical features after etching.

Table 3: Independent t-test for SBS means (MPa) between adhesive types.								
	n	Mean	<i>t</i> -value	sig	Std. deviation	Std. error mean		
Zirconia								
Control								
Assure [®] Plus	10	3.3039	0.961	0.349	0.31672	0.10016		
Transbond [™] XT	10	3.1865	0.961		0.22104	0.06990		
HF								
Assure [®] Plus	10	4.0989	1.413	0.175	0.14546	0.04600		
Transbond [™] XT	10	4.0182	1.413		0.10698	0.03383		
Al ₂ O ₃								
Assure [®] Plus	10	17.6450	20.951	0.000*	0.25779	0.08152		
Transbond [™] XT	10	15.0527	20.951		0.29435	0.09308		
SBS: Shear bond strength, HF: Hydrofluoric acid, Al ₂ O ₃ : Aluminum oxide, MPa: Megapascal, sig.: Statistical significance, *: significant difference.								

While Quentin *et al.*^[6] found that the used of high concentration of HF 40% is appropriate for conditioning of zirconia specimens because it leads to uniform and fast etching. Furthermore, our result is in contrast with Zhang *et al.*^[5] who considered HF acid as a promising surface conditioning method to promote bracket-zirconia bonding without excessive zirconia damage. However, intraoral etching by HF can be dangerous and considered toxic. Hence, alternatives to HF can be used like orthophosphoric acid, sandblasting, and carbon dioxide laser.^[27,28]

Several researchers have sandblasted zirconia specimens with Al_2O_3 particles to provide higher mechanical retention^[9,10,16,29] by increasing the surface roughness of zirconia.^[30] Farag^[31] used Al_2O_3 particle sizes for sandblasting (40, 80, and 110 μ m) and observed that the use of coarser Al_2O_3 particles led to an increase in surface irregularities and then increased the surface area available for adhesive, improving the micromechanical retention and finally increasing the bond strength values.

The effectiveness of sandblasting in increasing the SBS between the bonding materials and the zirconia specimens is similar to the study of Ourahmoune *et al.*^[32] They showed that air abrasion increases surface roughness and wettability of the zirconia materials, and the contact angle increases, increasing the mechano-retention and enhancing the bond strength.

Table 4: Distribution of ARI scores among groups.								
Group	0	1	2	3				
Assure [®] Plus								
HF	4	6	0	0				
Control	5	5	0	0				
Al_2O_3	0	1	6	3				
Transbond [™] XT								
HF	3	7	0	0				
Control	9	1	0	0				
Al_2O_3	1	5	4	0				
ARI: Adhesive remnant index, HF: Hydrofluoric acid, Al ₂ O ₃ : Aluminum								

AKI: Adhesive remnant index, HF: Hydrofluoric acid, Al₂O₃: Aluminum oxide

Mehta *et al.*^[16], in their study, concluded that bonding brackets to sandblasted zirconia surfaces with Reliance Assure Plus resulted in higher SBSs than the retention between the orthodontic attachment and the adhesive.

Kwak *et al.*^[1] observed that when air abrasion was done with $30 \,\mu m \, Al_2O_3$ on the glazed zirconia, producing a randomized rough surface and providing acceptable bonding of metal bracket to glazed zirconia.

The values of SBS obtained by Assure[®] Plus and Transbond[™] XT with zirconia specimens did not differ significantly in the control and HF groups. In contrast, the SBS of orthodontic brackets bonded to zirconia using Assure[®] Plus was significantly higher than those bonded by Transbond[™] XT for Al₂O₃ groups because of the higher flowability of Assure[®] Plus, which provides adequate SBS. The same conclusions were concluded by Amirabadi *et al.*^[33], wherein the adhesion of the orthodontic bracket to ceramic that bonded by Assure[®] Plus was significantly superior to that when bonded with Transbond[™] XT.

In contrast to these studies, Mehta *et al.*^[16] reported a similar bonding strength of Assure[®] Plus and TransbondTM XT for zirconia specimens. Furthermore, Douara *et al.*^[29] revealed no significant differences between the use of TransbondTM XT and Assure[®] Plus on zirconia specimens.

ARI was used to determine the position and mode of adhesive failure. Several studies have advocated that it is preferable for the occurrence of adhesion failure at the tooth adhesive interface so that the resin remnants on the surface can be cleaned safely with rotary instruments.^[30,34-36]. When debonding orthodontic brackets from the enamel surface, it is important to avoid enamel damage and with minimal adhesive remaining on the teeth surface. Likewise, for all restorations, the aim is for the debonded area to have minimal cohesive damage to ceramic or zirconia and, at the same time, have minimal residual adhesive left.^[16]

The adhesive failure in the control and HF group bonded by TransbondTM XT or Assure[®] Plus had a score of 0 and 1, which was designated to adhesivezirconia interface failure. Contrarily, most of the models in the Al₂O₃ group

Table 5: Descriptive statistics and Kruskal–Wallis test of ARI.										
Zirconia	Primer type	Group	n	Minimum	Maximum	Mean	Std. deviation	Kruskal-Wallis Test		
models								Chi-square	Df	Asymp. Sig.
	Assure [®] Plus	Control	10	0.00	1.00	0.3000	0.48305	19.262	2	0.000
		HF	10	0.00	1.00	0.5000	0.52705			
		Al_2O_3	10	1.00	3.00	2.0000	0.66667			
	Transbond [™] XT	Control	10	0.00	1.00	0.1000	0.31623	14.783	2	0.001
		HF	10	0.00	1.00	0.3000	0.48305			
		Al_2O_3	10	1.00	2.00	1.5000	0.52705			
ARI: A desire remnant index HE: Hydrofluoric acid ALO: Aluminum oxide DE: degree of freedom. Asymp Sig : significant difference at $P < 0.05$										

bonded with Assure[®] Plus had a score of 2 and 3, which was designated to adhesivebracket interface failure, while in Al_2O_3 group bonded with TransbondTM XT had a score of 1 and 2, which was designated as mixtype, indicating a favorable failure mode. This result suggests that the TransbondTM XT is a suitable adhesive for use with zirconia material.

Limitation of the study

These *in vitro* studies were applied to evaluate the effect of two types of adhesive material and two surface treatment methods on SBS, but the effect of other factors that intervene in the oral environment was not considered in our investigation. These contributing variables affect the SBS values in the oral environment, such as the pH level of saliva, complex microflora, temperature, stress generated by the orthodontic archwire, and masticatory force.

CONCLUSION

The SBS obtained when bonding metal orthodontic brackets using the TransbondTM XT adhesive or Assure[®] Plus with air abrasion by Al_2O_3 particles were satisfactory for zirconia restoration. On the other hand, inadequate SBS values were achieved when using HF treatment of zirconia surface, so acid etching of zirconia models by HF had no significant effect on SBS of metal brackets bonded to zirconia specimens. According to the ARI result, TransbondTM XT is a suitable adhesive for use with zirconia material.

Ethical approval

The research/study was approved by the Institutional Review Board at the research ethics committee of the College of Dentistry/Mosul University, number UoM.Dent.23/33, dated 4th June 2023.

Declaration of patient consent

Patient's consent was not required as there are no patients in this study.

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Nil.

Conflicts of interest

There are no conflicts of interest.

Use of artificial intelligence (AI)-assisted technology for manuscript preparation

The authors confirm that there was no use of artificial intelligence (AI)-assisted technology for assisting in the

writing or editing of the manuscript, and no images were manipulated using AI.

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