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Comparison of shear bond strength of metallic brackets bonded to ceramic surfaces utilizing different adhesive systems: An *in vitro* study

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Abstract

OBJECTIVE: To compare the shear bond strength (SBS) of orthodontic brackets bonded to three different types of ceramic surfaces (feldspathic, lithium disilicate, and zirconium) using Assure® Plus All and Transbond™ XT adhesives.

MATERIALS AND METHODS: The sample comprised 72 monolithic computer-aided design and computer-aided manufacturing (CAD/CAM) ceramic samples that were randomly divided into six groups of 12 specimens each. Three groups (G1, feldspathic ceramic; G3, lithium disilicate ceramic; G5, zirconium surfaces) were bonded to metal brackets using Assure® Plus All adhesive, whereas the remaining three groups (G2, G4, G6; with the ceramic type in the same order as that in the previous groups) were bonded to metal brackets using Transbond™ XT. The samples were then subjected to 10,000 thermocycles. The SBS was calculated using the shear tests. The site of bonding failure was classified using the adhesive remnant index (ARI) score. One-way analysis of variance (ANOVA) and Kruskal–Wallis tests were used for statistical analyses at a 5% significance level.

RESULTS: Statistically significant differences were observed in the mean SBS values of the groups ($P < 0.001$). The mean SBS for G6 (zirconium plus Transbond™ XT) (2.52 MPa) was significantly lower than that for all other groups. Furthermore, statistically significant differences were found in the ARI score distribution among the groups ($P < 0.001$). Differences were identified between G6 and G3 (lithium disilicate Plus All Assure® Plus All) and G5 (zirconium plus Assure® Plus All).

CONCLUSIONS: The mean bonding strength of brackets with Assure® Plus All was higher than that with Transbond™ XT for all three types of ceramics. However, all groups, except the zirconium plus Transbond™ XT group, showed acceptable bonding strength for orthodontic purposes. The application of hydrofluoric acid followed by silane and finally the Assure® Plus All adhesive system is adequate for bonding brackets to any of the ceramic tested surfaces.

Keywords:

Dental ceramic, Micromechanical preparation, Orthodontic metallic brackets shear bond strength

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Introduction

Indirect ceramic dental restorations are considered the materials of choice in dentistry to replace lost or damaged teeth owing to their esthetic advantages, excellent mechanical properties, and biocompatibility.[1-3]

In recent years, the number of adult patients seeking orthodontic treatment has increased.[4–6] However, when orthodontic treatment is needed, difficulties may arise when bonding brackets to ceramic

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surfaces.[1,2,7,8] Conventional methods of bonding to the enamel may be ineffective for ceramic surfaces.[9] Therefore, successful adhesion protocols for materials are imperative, as it may be difficult to differentiate which restorative material is present in the oral cavity.[5]

Bracket bonding in orthodontics is temporary. Sufficiently high bond strength is required to resist masticatory and orthodontic forces, preventing bracket detachment during treatment. However, bond strength should allow the brackets to come off at the end of treatment without damaging the restoration surface, maintaining its initial structure.[3,4,6,10,11] *In vitro* studies have shown that bonding adhesives must be able to withstand loads of 5.9 to 7.8 MPa to be considered clinically suitable for orthodontic purposes.[12]

The Transbond™ XT adhesive system (3M Unitek, Monrovia, California, USA) is considered the most tested and compared orthodontic adhesive. Therefore, it has been used as a "control group" in many investigations comparing orthodontic adhesives.[10] The Assure® Plus All adhesive system (Reliance Orthodontic Products, Itasca, Illinois, USA) was recently introduced, with a specific indication for ceramic, amalgam, composite resin, and atypical enamel surfaces.[5,9,10]

The number of studies comparing the adhesives used in bonding brackets to ceramic surfaces is remarkable and is an extensively investigated topic. However, there are a limited number of investigations that had evaluated the effectiveness of bonding orthodontic brackets to ceramic surfaces using the Assure® Plus All and Transbond™ XT adhesives[10] and this is especially true for investigating the effectiveness of bonding brackets to Zirconia, and comparing this shear bond strength with the bonding strength of orthodontic brackets to other ceramic surfaces, namely; feldspathic ceramic, lithium disilicate. Furthermore, a similar study to ours performed by Naseh *et al*.[10] utilized ceramic discs instead of mimicking a ceramic tooth surface. Therefore, the present investigation aimed to evaluate the effectiveness of bonding orthodontic brackets to feldspathic ceramic, lithium disilicate ceramic, and zirconia surfaces using Assure® Plus All and Transbond™ XT adhesives.

Materials and Methods

Egas Moniz School of Health and Science, Portugal. The protocol of this study was approved by the scientific committee of the same school on the 17th of December 2019.

Sample size

The investigated model included 18 CAD/CAM ceramic blocks, each with four premolar buccal surfaces. The surfaces (n = 72 premolar buccal surfaces) were randomly partitioned into six groups, each containing 12 samples. The sample size calculation was computed based on a medium effect size of (0.5) for 80% power at a 5% significance level.

Ceramic blocks fabrication

The upper first premolar was scanned using a Vinyl Smart Optics scanner (Sensortechnik GmbH, Bochum, Germany). A three-dimensional builder program (Microsoft Corporation) was used to design the samples. The buccal surface of the scanned premolar was copied and added until four equal buccal surfaces were joined to one sample. Subsequently, the ceramic blocks were milled using the iCAM V5 smart digital software (imes-icore® GmbH, Eiterfeld, Germany) and a computer-aided design and computer-aided manufacturing (CAD/CAM) milling machine CORiTEC 250i (imes-icore® GmbH, Eiterfeld, Germany). A brush, water, and a polishing paste without fluoride were used to clean the ceramic blocks. Subsequently, the air was sprayed onto the specimens for 5 s.

Sample preparation

Groups 1 and 2 (G1 and G2) were assembled from feldspathic ceramic material (Vitablocs® Mark II, VITA Zahnfabrik, Bad Säckingen, Germany); groups 3 and 4 (G3 and G4) comprised lithium disilicate ceramic blocks (IPS e. max ® CAD, Ivoclar Vivadent AG, Schaan, Liechtenstein), and groups 5 and 6 (G5 and G6) comprised zirconia blocks (3M LavaTM Aesthetic, 3M Deutschland GmbH, Neuss, Germany).

Bonding/debonding the brackets

A layer of 9.6% hydrofluoric acid was applied to the ceramic surfaces of all groups for 2 min (Porc-Etch[TM]; Reliance Orthodontic Products, Itasca, Illinois, USA), rinsed, and dried for 30 s each. Subsequently, a layer of the Porcelain Conditioner silane was applied correspondingly for 1 min (Reliance Orthodontic Products), rinsed, and dried, following a previously reported protocol. Subsequently, the odd groups (G1, G3, and G5) were treated with the Assure® Plus All adhesive system, while the Transbond™ XT adhesive system was applied to the even groups (G2, G4, and G6) [Figure 1]. In all groups, the adhesive paste Transbond™ XT (3M Unitek) was applied over the base of MBT premolar metal brackets (Victory Series™; 3M Unitek). Subsequently, the brackets were positioned at the treated center of the ceramic surfaces. The same trained operator exerted a consistent force for 10 se to confirm uniform adhesive thickness. Thereafter, excessive adhesive was eliminated using a sharp scaler. Subsequently, the adhesive was photopolymerized using 3M Ortholux™ Luminous Curing light with a light intensity of 1,600 mW/cm² for 10 s from each of the mesial and distal sides at a distance of 3 mm from the bracket [Figure 1]. The specimens were maintained in distilled water (at 37°C) for 24 h in a Memmert® INE 400 universal incubator (Schwabach, Germany). Next, the samples were exposed to 10,000 thermal cycles in a Jukabo Labortech® thermocycler (Schabach, Germany). Each cycle lasted for 20 s in a cold bath (5°C), 20 s in a hot bath (55°C), and 20 s outside water at 37°C.

To determine the adhesive strength, each sample was subjected to shear bond strength (SBS) testing using a Shimadzu Autograph AG-IS Universal Testing Machine (Shimadzu Corporation, Tokyo, Japan) at a speed of 1 mm/min[13] until adhesive failure occurred. To ensure the stability of the specimens, each tested model was pre-installed on a customized self-curing acrylic resin block Schütz Futura Self (Schütz Dental Group, Rosbach, Germany). The acrylic blocks were then positioned and installed in the testing machine to sustain a parallel layout between the bracket base and the direction of the applied machine force. SBS values were calculated in megapascals (MPa) by dividing the force at debonding (N) by the surface area of the bracket base (mm^2).

The site of the bond failure was established using an optical microscope (Leica Microsystems Limited, Heerbrugg, Switzerland) at 10x magnification, and micrographs were taken using Leica Application Suite version 3.8.0 software (Leica Microsystems CMG, Switzerland). This procedure aimed to evaluate the amount of adhesive remaining on the ceramic surface and the base of the bracket, and consequently, to determine the site of adhesive failure. Thus, the adhesive remnant index (ARI)[14] was applied to categorize adhesive failure according to the amount of adhesive left on the ceramic surface after debonding as follows: Score "0" indicates that no composite resin was left on the tooth surface; score "1" indicates that less than 50% of the composite resin was still present; score "2" indicates that more than 50% of the composite resin was still present; and score "3" indicates that the entire amount of composite resin was still present on the tooth surface but bore a distinct imprint of the bracket base.[14]

Figure 1: Customized acrylic resin block to ensure tooth stability

Statistical analysis

IBM SPSS® Statistics v. 26 software (IBM, New York, USA) was used to perform descriptive and inferential statistical analyses. The Shapiro–Wilk test revealed that the SBS data were normally distributed, and Levene's test confirmed homoscedasticity. One-way ANOVA was used to compare the mean SBS values of the six groups, followed by Tukey's HSD test. The inferential analysis significance level was set at 5% ($p \leq 0.05$).

The nonparametric Kruskal–Wallis test was used to compare the distribution of ARI scores across the examined groups. Subsequently, pairwise comparisons with Bonferroni correction were performed. The inferential analysis significance level was set at 5% ($p \leq 0.05$).

Results

Six brackets were detached in G6 (Transbond™ XT adhesive applied to the zirconia surface) during the thermocycling stage, before applying the SBS test. Therefore, these samples were not included in the study, reducing the number of G6 samples to six valid specimens.

Table 1 displays the results of the descriptive analysis of mean SBS values for each sample group. The one-way ANOVA [Table 2] revealed a statistically significant

Table 1: Mean, standard deviation (SD), minimum and maximum and 95% Confidence Interval (95% CI) for the mean shear bond strength (SBS) values (MPa) recorded for the six examined groups

Group	*n*	Mean	SD	Min.-Max.	95% CI for Mean	*P*
G1	12	7.16	1.46	5.11-9.65	6.24-8.09	<0.001
G2	12	6.04	2.05	2.54-9.58	4.74-7.34	
G3	12	9.27	2.34	5.51-11.92	7.79-10-76	
G4	12	7.76	3.95	1.37-13.19	5.25-10.27	
G5	12	8.51	1.99	3.40-11.84	7.24-9.77	
G6	6	2.51	1.01	1.56-4.26	1.44-3.57	

Table 2: One way analysis of variance and the *P* value between and within the groups indicating the existence of statistically significant differences between the mean shear bond strength values of the groups (*P*<0.05)

	Sum of squares	df	Mean square	*Z*	*P*
Between groups	223,810	5	44,762	7,669	<0.001
Within groups	350,216	60	5,837		
Total	574,026	65			

difference between the mean SBS values of the six groups ($P < 0.001$). Using Tukey's analysis [Table 3], significant discrepancies were detected between G6 (Transbond™ XT adhesive applied to the zirconia surface) and all the other groups (G1–G5) except G2. The mean SBS for G2 (Transbond™ XT adhesive applied to feldspathic ceramic surfaces) was found to be significantly lower than the ones obtained for G3 (Assure® Plus All adhesive applied to lithium disilicate ceramic). Conversely, the remaining examined groups indicated statistically similar mean SBS values [Table 3].

Table 4 and Figure 2 illustrate the distribution of the ARI scores among the different groups. The majority of the specimens in G1, G2, G3, G4, and G5 had a mixed-type bonding failure (score "1" and score "2"), while none of the 72 models was assigned score 3 (all the adhesive remained on the ceramic surface). However, all samples in G6 (Transbond™ XT adhesive applied to zirconia surface) were assigned to score "0" (all the adhesive remained on the bracket mesh). The Kruskal–Wallis test verified the presence of statistically significant differences in the ARI score distribution among the groups ($p \leq 0.001$). Pairwise comparison [Table 5] revealed significant differences between G6 and both G3 (lithium disilicate Plus All Assure® Plus All) and G5 (zirconium plus Assure® Plus All).

Discussion

The realization of orthodontic treatment requires good adhesion between the bracket base and tooth surface to resist the forces of mastication, the forces imposed by the appliance, the PH, and the temperature changes in the oral cavity.[15] As dentistry evolves, new challenges are presented, such as the adhesion of brackets to restorative materials. The ideal bond must be strong enough to withstand the duration of orthodontic treatment and weak enough to allow debonding of the bracket at the end of treatment while maintaining the integrity of the ceramic surface.[16-18] Although the adhesion of ceramic restorations has advanced, failures still occur frequently, and this adhesion is commonly reported as insufficient.[19]

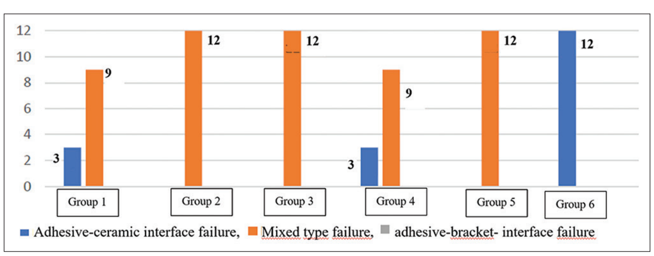

Figure 2: Types of adhesive failure among the examined groups

To meet our proposed objectives, an experimental *in vitro* study was designed. Hypothetically, *in vivo* studies are superior for testing material efficacy. Clinically, it is almost impossible to distinguish the adhesive potential of a new material without the influence of other variables that intervene in the oral cavity. Furthermore, clinical studies have found little appreciable variation in the effectiveness of treatments across these contexts.[20]

According to Faria *et al*.[21] variations in the composition of different types of ceramics produce distinct topographical characteristics after etching. Thus, hydrofluoric acid provides adequate adhesive strength on glass ceramics, but may not be effective on alumina- or zirconia-reinforced ceramics. In the same study, the authors suggested that hydrofluoric acid etching followed by silanization could be recommended as a protocol for the surface treatment of ceramics. Several previous studies have sandblasted ceramic surfaces with aluminum oxide particles to create greater mechanical retention.[5,9,10,22,23] However, this procedure and the use of a diamond bur to improve SBS can irreversibly damage the ceramic surface.[18,19] For this reason, Mehmeti *et al*.[18] recommend performing treatments that provide adequate bond strength while maintaining the integrity of the ceramic surface.

To submit the materials to extreme temperatures comparable to those found in the oral cavity, the samples were subjected to thermocycling. This laboratory procedure simulates material aging through temperature variations, which may influence adhesive strength.[15,19,21,24] Gale[25] suggested that 10,000 cycles correspond to one year of temperature changes in the oral cavity. Thus, for this investigation, we chose to simulate one year of aging of the samples, which is approximately equivalent to the minimum duration of an orthodontic treatment.

SBS tests are the most widely used in orthodontics to evaluate bond strength because of the similarity between this type of test and the forces performed during orthodontic treatment.[26] Standardization of the unit of measurement to express SBS values is of paramount importance to facilitate the comparison of results between studies. Thus, the most appropriate is the use of MPa or equivalent measures such as newton per square millimeter (N/mm^2) or meganewton per square meter (MN/m^2). These measures indicate the force per unit area required to move a bracket from its position.[26,27]

Table 3: Pairwise comparison between the mean SBS values of the six examined groups (*P*<0.008)

	G1-G2	G1-G3	G1-G4	G1-G5	G1-G6	G2-G3	G2-G4	G2-G5	G2-G6	G3-G4	G3-G5	G3-G6	G4-G5	G4-G6	G5-G6
P	0.865	0.281	0.990	0.748	0.004	0.021	0.508	0.141	0.052	0.646	0.970	0.001	0.974	0.001	<0.001

In conclusion, the mean SBS obtained using Assure® Plus All and Transbond™ XT with feldspathic and lithium disilicate ceramic surfaces did not differ significantly. In contrast, the SBS of orthodontic brackets bonded to zirconia surfaces using Transbond™ XT was significantly lower than that bonded with Assure® Plus All. However, a similar SBS among the three types of ceramics was observed when Assure® Plus All was used. As it is clinically difficult to determine which type of restorative material is present, it is pertinent to apply adhesives that are effective on various types of materials.[5,11]

The SBS values recorded using Assure® Plus All were higher than those recorded using Transbond™ XT for the three ceramic surface types. The mean SBS values in G2 (feldspathic treated with Transbond™ XT surface) and G4 (lithium disilicate conditioned by Transbond™ XT) were within the range of acceptable orthodontic reference values (the mean SBS values for G2 and G4 were 6.04 and 7.76 MPa, respectively). This is because SBS values between 5.9 and 7.8 MPa are proposed as clinically effective, allowing adequate adhesion of the bracket to the ceramic surface.[28-31] However, bond strengths exceeding 13 MPa may cause fractures on the ceramic surface during debonding of the bracket at the end of orthodontic treatment.[28,30,32] Six brackets in G6 were detached from the ceramic surface (brackets bonded to zirconia surfaces using the Transbond™ XT adhesive system) before the submission to the SBS test. The SBS test confirmed that the mean SBS value for this group (2.51 MPa) was significantly lower than the proposed optimal mean SBS values (5.9–7.8 MPa).

Our results align with those of Naseh *et al*.,[10] who reported significant differences in SBS among the examined groups. The highest mean SBS values were obtained for the lithium disilicate ceramic samples conditioned with the Assure® Plus All adhesive. The lowest was observed in feldspathic ceramic samples treated with Transbond™ XT Different ceramic surfaces may lead to distinct SBS values, as ceramics are diverse concerning their particle´s size and their crystalline form. It has been reported that lithium disilicate ceramic has less glass material than Feldspathic. Therefore chemical treatment with hydrofluoric acid creates a more porous pattern in the lithium disilicate, with increased retention due to the dissolution of the glass stage.[10] Also, the higher flowability of Assure® Plus All compared to Transbond™ XT might provide adequate bond strength. Similar conclusions were reported by Amirabadi,[22] wherein adhesion to feldspathic ceramics conditioned by Assure® Plus All was significantly superior to that when treated with Transbond™ XT.

Table 4: Distribution of ARI scores among the examined groups (results presented as median, inter-quartile range (IQR) and minimum-maximum)

Group	*n*	Median	IQR	Min.-Max.
G1	12	1.0	2	0-2
G2	12	1.0	0	1-1
G3	12	2.0	0	2-2
G4	12	1.0	2	0-2
G5	12	2.0	0	1-2
G6	6	0.0	0	0-0

Our and Amirabadi's[22] investigations observed superior adhesion to feldspathic ceramics conditioned by Assure® Plus All compared to feldspathic ceramics treated with Transbond™ XT. However, this superiority in adhesion was only statistically significant in the Amirabadi's[22] experiment. In contrast to these studies, Mehta *et al*.[5] reported a similar SBS between the application of Transbond™ XT and Assure® Plus All for feldspathic ceramic and zirconia specimens. However, later authors performed tensile tests rather than shear tests employed in our investigation. This is why it is not possible to compare the mean forces because the literature suggests that the values obtained in shear tests are usually higher than in tensile tests.[5] In contrast to the present study, Douara *et al*.[23] determined no statistically significant differences between the use of Assure® Plus All and Transbond™ XT on zirconia surfaces, but significant discrepancies between the groups treated with Assure® Plus All adhesive without prior application of silane. This finding suggests that the application of silane is crucial to achieve adequate adhesion between the bracket and zirconia, as silane promotes increased surface wettability.[28] Furthermore, several authors have reported that the use of hydrofluoric acid on zirconia surfaces is not sufficient to achieve good adhesion, as zirconia has a low silica content, which makes it acid-resistant, making it difficult to create porosities.[11,33,34] Despite this, in the present study, the use of Assure® Plus All provided acceptable adhesion values for the adhesion of brackets to zirconia surfaces.

The two aspects of adhesion involved in bonding a bracket are adhesive adherence to each bracket mesh and ceramic surface. In this investigation, ARI was used to determine the mode and position of adhesive failure. The assessment device was a 10x magnifier, which is comparable to the devices used in similar previous

Table 5: Pairwise comparison of ARI scores among the examined groups (Group 1 to Group 6)($P<0.008$). Significant differences between groups are denoted in bold

	G1-G2	G1-G3	G1-G4	G1-G5	G1-G6	G2-G3	G2-G4	G2-G5	G2-G6	G3-G4	G3-G5	G3-G6	G4-G5	G4-G6	G5-G6
*P**	1,000	0.020	1.000	0.120	0.055	0.011	1.000	0.074	0.090	0.020	1.000	**<0.001**	0.120	0.055	**<0.001**

studies. Our investigation found statistically significant differences between the adhesive failures among the examined groups. Several authors have advocated that it is preferable for adhesion failure to occur at the bracket-adhesive interface or within the adhesive so that the resin residue on the ceramic surface can be removed safely with rotary instruments.[30,35-38] Similarly, the adhesive failure in G2 (feldspathic conditioned by Transbond™ XT), G3 (lithium disilicate treated by Assure® Plus All), and G5 (zirconia conditioned by Assure® Plus All) was designated as mixed-type failures for all the specimens, indicating a favorable failure mode. Contrarily, all the samples in G6 (zirconia conditioned with Transbond™ XT) had a score "0" ARI designated to adhesive-ceramic interface failure. This result suggests that Transbond™ XT is not an unsuitable adhesive for use with zirconia. However, the ARI values obtained do not simply represent the SBS but also depend on other factors, such as the bracket mesh pattern and the type and thickness of the adhesive used. Thus, the SBS obtained may not be related to the type of ARI observed.[10]

A recent systematic review and meta-analysis[39] reported that the 71 articles included in their review were of high to medium risk of bias. The authors concluded that the most effective technique for bonding orthodontic brackets to ceramic surfaces depends on the type of ceramic used. However, recognizing the type of ceramic veneers or crowns in a clinical setting is not usually feasible. Therefore, using an effective surface treatment for all ceramic surfaces is a favorable and safer option. Based on the result of the present investigation, the protocol of applying a layer of hydrofluoric acid (9.6%) for two minutes, rinsed and dried, followed by application of a layer of silane for one minute, rinsed and dried, and finally, the Assure® Plus All adhesive system is recommended for bonding metallic brackets to all types of ceramic surfaces.

In the course of the research, some limitations emerged. Although a reasonable number of studies on ceramic bonding have been conducted, few similar investigations have reported on the use of Assure® Plus All on ceramic surfaces. Furthermore, no related standardized laboratory protocol exists, making it difficult to compare the results of the present study with those of previous studies. Finally, several factors that contribute to the oral environment were not considered in our investigation. Despite the benefits of *in vitro* studies applied to evaluate specific materials, several contributing variables in the oral cavity environment that influence the SBS values were not considered, namely variations in the intraoral pH level, temperature, stress generated by the orthodontic arch wires, and complex microflora existing in the oral cavity.

Conclusions

The bond strengths obtained when bonding metal orthodontic brackets using the Assure® Plus All and Transbond™ XT adhesive systems were satisfactory for feldspathic ceramics and lithium disilicate ceramics, although the values achieved with Assure® Plus All were higher. This was not the case for zirconia, for which inadequate bond strength values were achieved using the Transbond™ XT adhesive system. The application of hydrofluoric acid followed by silane and finally the Assure® Plus All adhesive system is adequate for bonding brackets to any of the tested surfaces.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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