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Quick Response Code:
Website:
www.jorthodsci.org
DOI:
10.4103/jos.jos_79_22

The effect of ceramic surface conditioning on bond strength of metallic brackets: An *in vitro* study

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Abstract

OBJECTIVE: To compare the shear bond strength (SBS) of brackets bonded to three different types of ceramic surfaces (feldspathic ceramic, lithium disilicate ceramic, and zirconia), conditioned with either hydrofluoric acid or sandblasting, using Assure® Plus All bonding agent.

MATERIALS AND METHODS: A total of 72 monolithic CAD/CAM ceramic specimens were divided into six groups of 12 samples. Three groups (G1: feldspathic ceramic, G3: lithium disilicate ceramic, G5: zirconia surfaces) were conditioned with 9.6% hydrofluoric acid, while the remaining three (G2, G4, G6; with ceramic type in the same order as the previous three groups) were prepared with 50 µm aluminum oxide sandblasting. Premolar brackets were bonded using light-cured Assure® Plus All. The SBS and adhesive remnant index (ARI) were recorded and submitted to inferential analysis using one-way analysis of variance and Kruskal–Wallis tests, respectively. The significance level was set at 5% ($P \leq 0.05$).

RESULTS: The mean SBS values for the three different ceramic groups conditioned with hydrofluoric acid (G1: 7.2 ± 1.5 MPa, G3: 9.3 ± 2.3 MPa, G5: 8.5 ± 2.0 MPa) were significantly higher than those obtained for the groups prepared by sandblasting before bonding (G2: 7.5 ± 1.8 MPa, G4: 4.4 ± 2.0 MPa, G6: 4.3 ± 2.8 MPa).

CONCLUSIONS: The hydrofluoric acid treatment produced a favorable SBS for all three examined ceramic types before bracket bonding with Assure® Plus All. In comparison, sandblasting yielded a satisfactory SBS only with feldspathic surfaces. Furthermore, the ARI indicated a higher frequency of mixed-adhesive failures except for lithium disilicate conditioned with sandblasting. Therefore, using hydrofluoric acid is likely to be especially recommended when the clinician is not aware of the brand of ceramic restorative material.

Keywords:

Ceramic, micromechanical preparation, orthodontic metallic brackets, shear bond strength

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Submitted: 02-Sep-2022
Revised: 23-Oct-2022
Accepted: 16-Jan-2023
Published: 04-Sep-2023

Introduction

Ceramics have become the most appealing materials for indirect dental restorations due to their superior strength and aesthetic characteristics.[1] They are increasingly common in adult patients seeking orthodontic treatment and therefore there is a growing need to bond brackets to pre-existing ceramic surfaces.[2,3] The success of orthodontic treatment depends on an adequately resistant and robust adhesion between the brackets and the designated surfaces. Numerous concerns may arise when bonding brackets to ceramic restorations, such as which bonding protocol to follow based on the ceramic composition and how to secure the aesthetic and functional viability of the ceramic surface following bracket debonding. Such questions arise frequently and represent a real challenge in orthodontic clinical

How to cite this article: Ferreira R, Pereira PM, Pitschieller R, Proença L, Bugaighis I. The effect of ceramic surface conditioning on bond strength of metallic brackets: An *in vitro* study. J Orthodont Sci 2023;12:42.

practice, not least because the clinician is rarely aware of the type of ceramic material used in the restoration.[2–4]

In vitro investigations are frequently used to assess the shear bond strength (SBS) and the adhesive remnant index (ARI).[3,5,6] Bracket bonding to a ceramic surface is deemed successful when the SBS ranges between 6 and 8 MPa.[7] The SBS of ceramic-bonded brackets is affected by three factors: the ceramic material, the micromechanical, or chemical preparation of the ceramic surface before the bonding process[4] Several categories of ceramics are used for dental restorations, the most frequently employed being feldspathic, lithium disilicate and zirconia.[8] The ARI is a qualitative system used to determine the residue of the adhesive left on the tooth after debonding. It is a critical element to be deemed in the selection of the used orthodontic adhesive.[9]

Ceramics are categorized according to their reaction to hydrofluoric acid to acid-sensitive materials, such as feldspathic and lithium disilicate, or acid resistant, such as zirconia, depending on their reaction to hydrofluoric acid. Commonly, ceramic micromechanical preparation is performed by applying hydrofluoric acid. Hydrofluoric acid etching causes increased ceramic surface roughness, creating undercuts and increasing the superficial area available for bonding. Subsequently, the application of a silane coupling agent enhances the wettability of the etched ceramic surface and establishes a covalent chemical bond between the silica of the ceramic restoration and the organic component of the bonding resin.[3] Another possible method of micromechanical preparation is blasting at high pressure using aluminum oxide.[10–12]

A recently introduced universal adhesive (Assure® Plus All surface-bonding resin) is claimed to have a sufficient SBS to bond brackets to ceramic surfaces. The producer recommends sandblasting the surface before applying the adhesive. However, in clinical practice, etching with hydrofluoric acid is the most-used micromechanical preparation method. It has, therefore, become imperative to compare these two preparation methods.[10–12]

Several previous studies explored the effectiveness of micromechanical and chemical conditioning techniques on the SBS of ceramic-bonded brackets.[10–12] However, an evaluation of micromechanical ceramic conditioning techniques (hydrofluoric acid etching or sandblasting) using Assure® Plus All has not been made. Therefore, this study aimed to identify the most efficient of two methods (hydrofluoric acid or sandblasting) for conditioning the three types of ceramic surface frequently used in dentistry (feldspathic, lithium disilicate, and zirconia), before bonding a bracket using the adhesive system Assure® Plus All.

Materials and Methods

This experimental *in vitro* investigation was granted ethical approval by the Ethics Committee of Egas Moniz School of Health and Science.

Sample size

The experimental model comprised 18 ceramic blocks embedded in resin cylinders, each block had four vestibular premolar surfaces. The total sample (72 premolar vestibular surfaces) was divided into six groups, each of 12 specimens. The sample size was determined based on an estimated medium effect size (0.5) for an 80% power at a 5% significance level.

Sample preparation

Groups 1 and Group 2 (G1, G2) comprised feldspathic ceramic blocks (Vitabloc® Mark II, VITA Zahnfabrik, Bad Säckingen, Germany). Groups 3 and Group 4 (G3, G4) consisted of lithium disilicate ceramic blocks (IPS e.max® CAD, Ivoclar Vivadent AG, Schaan, Liechtenstein), while Groups 5 and Group 6 (G5, G6) were assembled from zirconia blocks (3M LavaTM Esthetic, 3M Deutschland GmbH, Neuss, Germany). For sample fabrication, firstly, a premolar was scanned with a Computer Aided Design/Computer Aided Manufacturing (CAD/CAM) Vinyl scanner (Smart Optics Sensortechnik, GmbH, Bochum, Germany). Subsequently, a digital model with four buccal surfaces was created using a three-dimensional digital builder program (Microsoft Corporation). The ceramic blocks were then milled using iCAM V5 (imes-icore® GmbH, Eierfeld, Germany) and a CAD/CAM milling machine CORiTEC 250i (imes-icore). The ceramic blocks were cleaned using a brush and polishing paste without fluoride and water. Subsequently, they were rinsed thoroughly with water and dried by using an air spray for 5 seconds.

Bonding/debonding the brackets

G1, G3, and G5 ceramic surfaces were micromechanically prepared by applying a layer of 9.6% hydrofluoric acid for 2 minutes (Porc-Etch™; Reliance Orthodontic Products, Itasca, Illinois, USA), rinsed, and dried each for 30 seconds. In this order, the corresponding surfaces in G2, G4, and G6 were conditioned by sandblasting with 50 µm aluminum oxide particles (EtchMaster®; Reliance Orthodontic Products) at a pressure of 40 psi for 3 seconds at a distance of 1–2 mm, rinsed and dried each for 30 seconds. A layer of the adhesive system Assure® Plus All (Reliance Orthodontic Products) was utilized and then dried carefully in all groups, preceded by the application of a layer of the Porcelain Conditioner silane for one minute (Reliance Orthodontic Products), then rinsed and dried following the same above-reported protocol. MBT premolar metal brackets (Victory Series™; 3M Unitek) were used. In all groups, the adhesive paste Transbond™ XT (3M Unitek) was applied over the base of the brackets where they were positioned at the treated center of the samples. A consistent force was exerted by the same trained operator for 10 seconds to confirm uniform adhesive thickness. Thereafter, the excessive adhesive was eliminated using a sharp scaler. Subsequently, the adhesive was photopolymerized (with a 3M Ortholux™ Luminous Curing light) with a light intensity of 1,600 mW/cm², for 10 seconds from each of the mesial side and distal side at a distance of 3 mm from the bracket. All the brackets were bonded by the same operator on the same day.

The sample was then stored in distilled water at 37°C for 24 h in a universal Memmert INE 400 incubator (Schwabach, Germany). Later, the sample was subjected to 10,000 thermal cycles in a Jukabo Labortech® thermocycler (Schabach, Germany), with each cycle comprising 20 seconds in a cold bath (5°C) followed by another 20 seconds in a hot bath (55°C) and a final 20 seconds out of the water at the room temperature.[13–17]

Each test was performed individually using a Universal Autograph AG-IS test machine (Shimadzu Corporation). Every tested case was pre-installed on a customized acrylic resin block (Schütz Futura Self)[18] to ensure the stability of the block [Figure 1]. The acrylic blocks were placed and fixed in the testing machine to maintain a parallel arrangement between the bracket base and the path of the applied machine force.[19] Flattened steel rod applied the shear force to the bracket at a crosshead velocity of 1 mm/min. The SBS values were computed in megapascals by dividing the force at fracture (N) by the surface area of the bracket base (mm²). The location of the bond failure was determined under an optical microscope (Leica MZ6) at 10× magnification, and the ARI[20] was used to establish the amount of adhesive residue on the tooth surface after debonding. The ARI grouping was based on the following scoring system: score "0," no composite resin remained on the tooth surface; score "1," <50% of the composite resin was left on the tooth surface; score "2," more than 50% of composite resin remained on the tooth surface; score "3," all the composite resin was left on the tooth surface with a marked imprint of the bracket base.[6,9]

Figure 1: A customized acrylic resin block to ensure the tooth's stability

Statistical analysis

Descriptive and inferential data analyses were carried out using IBM Statistical Package for the Social Sciences v. 26. The fit of the SBS data to normality and homoscedasticity were assessed and confirmed. One-way analysis of variance (ANOVA) was undertaken to compare the mean SBS values among the examined groups, followed by a post-hoc Tukey HSD test for pairwise multiple comparisons. The ARI scores were compared across the examined groups using the Kruskal–Wallis test, followed by multiple comparisons, with Bonferroni correction. The significance level for the inferential analyses was set at 5% ($P \leq 0.05$).

Results

Two brackets were detached from G4 and one bracket from G6 (conditioned with sandblasting) before starting the SBS test. Therefore, they were removed from their allocated experimental groups.

Table 1 presents the results of the descriptive analysis of the mean SBS values for each group. The one-way ANOVA results are displayed in Table 2, indicating the existence of statistically significant differences between the mean SBS values of the groups ($P < 0.05$). Post-hoc analysis [Table 3] revealed that G4 (sandblasted lithium disilicate) exhibited significantly lower mean

Table 1: Mean, standard deviation (SD), interval, minimum and maximum, and 95% Confidence Intervals (95% CI) of the SBS values (MPa) recorded for the six examined groups. *P* value revealed by One-way ANOVA

Group	*n*	Mean	SD	95% CI		*P*
				Minimum	maximum	
Group 1	12	7.16	1.46	5.11	9.55	<0.001
Group 2	12	7.48	1.84	3.94	9.55	
Group 3	12	9.27	2.34	5.51	11.92	
Group 4	10	4.41	1.99	1.67	8.52	
Group 5	12	8.51	1.99	3.40	11.84	
Group 6	11	4.26	2.78	1.30	10.23	

Table 2: One-way ANOVA and the *P* value between and within the groups indicating the existence of statistically significant differences between the mean SBS values of the groups (*P*<0.05)

	Sum of squares	df	Mean square	*Z*	*P*
Between groups	241.76	5	48.35	10.98	<0.001
Within groups	277.57	63	4.41		
Total	519.34	68			

Table 3: Pairwise comparison between the mean SBS values of the six examined groups at $P<0.008$

Inter-group	1–2	1–3	1–4	1–5	1–6	2–3	2–4	2–5	2–6	3–4	3–5	3–6	4–5	4–6	5–6
P	0.999	0.151	0.037	0.621	0.018	0.304	0.014	0.836	0.006	0.0001	0.947	0.0001	0.0001	1.000	0.0001

Table 4: Distribution of the type of failure according to the ARI scores among the three examined groups

Group	ARI score			
	Score 0	Score 1	Score 2	Score 3
Group 1	3	6	3	0
Group 2	1	3	8	0
Group 3	0	0	12	0
Group 4	11	1	0	0
Group 5	0	2	10	0
Group 6	2	6	4	0

SBS values ($P \leq 0.05$) than the other groups, except for G6 (sandblasted zirconia) ($P = 1.00$). Moreover, there was a significant discrepancy between G6 (sandblasted zirconia) and the remaining groups, except G4 (sandblasted lithium disilicate) ($P = 1.00$). No significant differences were observed among the mean SBS values from the other groups ($P < 0.05$).

Table 4 and Figure 2 present the distribution and frequency of ARI scores among the six groups. The most frequently assigned score across all groups was "2" (mixed-type bonding failure), while none of the groups had an ARI score of "3" (all adhesive remains on the ceramic surface). A Kruskal–Wallis test confirmed the existence of statistically significant differences among the groups ($P \leq 0.001$). Pairwise comparison [Table 5] confirmed the presence of significant discrepancies ($P \leq 0.008$) between G4 (sandblasted lithium disilicate) and each of the following groups: G1 (feldspathic treated with hydrofluoric acid), G5 (zirconia conditioned with hydrofluoric acid), and G6 (sandblasted zirconia). However, there were no significant differences in the distribution of the ARI between the other groups ($P > 0.008$).

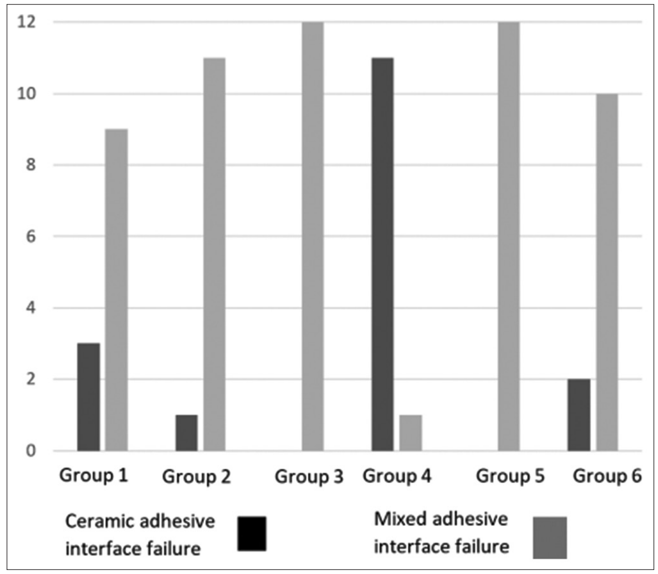

Figure 2: Displaying the type of adhesive failure among the groups

Discussion

The present *in vitro* study compared the SBS of orthodontic brackets bonded to feldspathic porcelain, lithium disilicate, and zirconia surfaces, prepared with hydrofluoric acid or aluminum oxide blasting, using Assure® Plus All. Theoretically, laboratory-based investigations are not genuinely illustrative of the oral environment due to the multiple confounding factors in the oral cavity that might cause a failure of bracket bonding. *In vivo* factors that might have an effect on bonding include pH variation, the cyclic mastication load, and temperature fluctuations. However, clinical research has shown few significant differences in treatment efficiency between both settings.[11,20,21]

Samples were immersed in distilled water at 37°C for 24 h to induce hydrolytic degradation at the adhesive interface. Then, they were immediately placed in the thermocycling machine, as recommended by ISO standard 29022:2013.[17] Thermocycling aims to simulate thermal stress to which dental materials and teeth are exposed in the mouth, compressing years of natural aging into a much shorter time. Although thermocycling is considered an inevitable regime undertaken in *in vitro* experiments, the ample investigations published in this area failed to agree on an integrated protocol concerning thermocycling application. This heterogeneity in methodology makes it difficult to compare the outcome of similar investigations.[14,15,17,22] Similarly to orthodontic treatment, excess thermal cycles can negatively affect the bond strength value and we, therefore, used 10,000 thermal cycles in the present study. It has been assumed that temperatures between 0°C and 68°C are consistent with the minimum and maximum temperatures recorded in the oral cavity. Accordingly, we chose 5°C and 55°C as the temperature extremes in this study: samples spent 20 seconds in a cold bath, 20 seconds in a hot bath, and then 20 seconds out of the water at room temperature.[14,15,17]

The groups of bonded brackets were subjected to a shear test using the universal testing machine to examine and evaluate their bond strength. The force applied was parallel to the long axis of the specimen and was submitted as close as possible to the adhesive-bracket interface. A customized loop designed exclusively for this type of

Table 5: Pairwise comparison among the ARI scores as revealed by the Kruskal–Wallis test with Bonferroni correction (at $P<0.008$)

Inter-group	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	0.068	0.068	0.013	0.009	0.625	1.00	0.001	0.440	0.181	0.001	0.440	0.181	0.001	0.003	0.034

test was fitted to the cervical wings of the brackets[9] The shear test speed was 1 mm/min, as recommended.[17]

To achieve adequate SBS, it is essential to prepare the ceramic surface before bonding. The conditioning method can be chemical, using hydrofluoric acid, or mechanical, using a technique such as sandblasting with aluminum oxide.[8-10] We used both methods in this study, with the chemical conditioning achieved with 9.6% hydrofluoric acid.[12] The adhesion strength values observed for the three types of ceramic when prepared with hydrofluoric acid were higher than the corresponding figures observed with sandblasting preparation. The mean SBS values in the shear test in groups G1, G2, G3, and G5 were within the range of minimum adhesion force values for bonding orthodontic brackets to ceramics (6–8 MPa).[4,23] On the other hand, G4 and G6 had low mean adhesion strength values leading to debonding of two and one bracket(s), respectively, before submitting the groups to the SBS test.

Eliasson and Dahl[22] concluded that applying hydrofluoric acid is an effective means of conditioning ceramic surfaces before bonding. It increases the mechanical retention of the bracket by the preferential dissolution of the ceramic vitreous phase. In another investigation, in contrast, Juntavee *et al*.[4] reported that ceramic conditioning with 9.6% hydrofluoric acid resulted in a low SBS value compared to other preparation techniques such as sandblasting with 50 μm aluminum oxide particles. Therefore, it is difficult to derive a consensus from previous studies, probably due to the heterogeneity of their methodology and applied protocols.[10,20] However, in a systematic review of 45 *in vitro* articles, Bach *et al*.[2] recommended applying silane following conditioning with 9.6% hydrofluoric acid to enhance adhesion strength. In this study, 9.6% hydrofluoric acid was used as a chemical etching agent followed by the application of silane coupling.

Bonding between silica-based ceramics and resin cements can be produced with the use of a silane coupling agent. Silane molecules interact with water to produce three hydroxy-silyl groups (-Si-OH) from the respective methoxy-silyl groups (-Si-O-CH_3).[24] The silanol groups later interact to produce a siloxane (-Si-O-CH_3) link with the silica on silica-based ceramics.[23,25] The methacryloyl groups of the silane molecules interact with the methacryloyl groups of the adhesive resin in a vigorous polymerization process.[24,25]

We observed that both preparation methods (hydrofluoric acid and sandblasting) improved the feldspathic-resin SBS. Several researchers have reported that such an enhancement of adhesion occurs due to the presence of a silica-based matrix (acid sensitive). This is because hydrofluoric acid reacts with the glassy phase of the material, causing the dissolution of the feldspathic surface layer and exposing the silica (a base component of the glassy phase) for chemical bonding with silane.[26-28]

We noticed that lithium disilicate ceramic surface etching with hydrofluoric acid was more efficient than sandblasting. Similarly to feldspathic ceramics, the silica-based matrix of lithium disilicate ceramics is acid-sensitive and therefore susceptible to the action of hydrofluoric acid. However, it is also composed of crystalline lithium disilicate particles, which are arranged in an intercalated, randomly oriented matrix, and therefore hydrofluoric acid is unlikely to produce a consistent topographical change in the surface.[29,30]

In the present study, treating zirconia with hydrofluoric acid proved to be an adequate method of micromechanical surface preparation. Several investigators[31–37] consider zirconia to be an acid-resistant material due to its polycrystalline structure and demonstrate the effectiveness of sandblasting the zirconia surface in association with a chemical preparation. While others[25,38] claim that hydrofluoric acid can change zirconia topography by reacting with its crystalline particles, causing them to dissolve gradually and shrink in size. In this study, only hydrofluoric acid conditioning before bonding with Assure® Plus All was effective. The sandblasting procedure did not produce satisfactory results when used with a 10-MDP-based adhesive, i.e., Assure® Plus All.[39]

Mode of bracket failure

Bonding bracket to a ceramic restoration has two adhesion facets: adhesion of the cement to the treated ceramic surface and adhesion of the cement to the bracket mesh. The ARI was used to evaluate the amount of adhesive remaining after the detachment of the brackets and to characterize the mode of adhesive failure. The assessment tool was a 10× magnifier, similar to that used in previous studies.[3,11,20,21] When detaching brackets from an enamel surface, adhesive failure occurring at the tooth-adhesive interface is preferable because it does not result in adhesive residue on the enamel surface, which is an advantage in the clinical environment. However, a mixed-type adhesive failure is recommended with adhesive bonding to a ceramic surface to avoid ceramic fracture during debonding. Subsequently, a rotary instrument can remove the remaining adhesive on the enamel surface. However, adhesive failure might not be due to inadequate adhesion strength alone. Other factors could also compromise bonding strength, such as bracket base configuration, elastic modules, increased adhesive thickness, and the composition of the used adhesive.[3,4,40] In our study, most specimens had a mixed-type failure among the examined groups except for lithium disilicate prepared with sandblasting (G4) where the adhesive failure was mainly at the ceramic-adhesive failure. This result indicates that sandblasting should not be an option for preparing lithium disilicate restorations before bonding. On the other hand, a similar occurrence of mixed-type failure was noticed in feldspathic groups (G1 and 2) and in zirconia groups (G 5 and 6) prepared with hydrofluoric acid or sandblasting, respectively. This might suggest that feldspathic and zirconia could be conditioned successfully by either sandblasting or hydrofluoric acid.

Therefore, our study proposes using hydrofluoric acid before bonding with Assure® Plus All adhesive especially when the type of the ceramic restoration is not known. More *in vivo* studies are recommended to confirm our findings in a clinical setting.

The scarcity of investigations similar to the present study and the heterogeneity of the protocols used with the Assure® Plus All system limit the scope for a critical comparison of our results. However, our study expands the horizon for new research in the field by providing a clear protocol to be followed and a set of benchmark results for future comparison.

Conclusions

The hydrofluoric acid treatment produced a favorable SBS for all three examined ceramic types (feldspathic, lithium disilicate, and zirconia) before bracket bonding with Assure® Plus All. In comparison, sandblasting yielded a satisfactory SBS only with feldspathic surfaces. Furthermore, the ARI revealed a favorable mixed-type adhesive failure for the three types of ceramic surfaces except the lithium disilicate group conditioned with sandblasting. Therefore, using hydrofluoric acid is likely to be especially recommended when the clinician is not aware of the brand of ceramic restorative material.

Financial support and sponsorship
Nil.

Conflicts of interest
There are no conflicts of interest.

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