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ORIGINAL RESEARCH

EFFECT OF THREE SURFACE TREATMENTS ON SHEAR BOND STRENGTH OF METAL BRACKETS TO LAYERED ZIRCONIA

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ABSTRACT

Background: Orthodontic bonding has evolved from Buonocore's acid-etching technique, allowing direct bonding, especially crucial in adult orthodontic treatment and aesthetic dental care. Ceramics, notably zirconium-based materials, are favoured for their aesthetics and mechanical properties. However, achieving strong adhesive bonds to composite or ceramic restorations while minimising bond failure remains challenging, with mechanical enamel etching risking restoration integrity. Combining chemical treatments with silane application is proposed to enhance adhesion while preserving ceramic integrity.

Methods: Fifty-two zirconia discs were randomly allocated to four groups: control, diamond burs, sandblasting, and 9.6% hydrofluoric acid (HFA). After surface treatment, discs were coated with a silane primer, cured, and bonded with pre-adjusted stainless steel brackets. A universal testing machine was employed to measure each specimen's shear bond strength (SBS), while scanning electron microscopy (SEM) was employed to analyse their topography. Post-debonding, the adhesive residue and ceramic surface condition were assessed using the Adhesive Remnant Index (ARI) and the Porcelain Fracture Index (PFI). Ethical approval was obtained from the Institutional Review Board (SRMDC/IRB/2021/MDS/NO.101).

Results: Sandblasting and HFA treatment showed significantly higher mean SBS (14.94 MPa and 13.94 MPa, respectively) compared to diamond bur treatment (8.38 MPa) and the control group (3.21 MPa). SEM analysis revealed effective surface roughening in sandblasted and HFA-treated groups. Statistical analysis demonstrated significant differences in SBS between the groups and distribution of ARI and PFI scores across treatments.

Conclusion: Combining silane with mechanical or chemical roughening enhances SBS, with sandblasting and HFA being effective. However, mechanical roughening risks ceramic damage, highlighting the need for careful surface preparation. Adhesive failures that occur primarily at the ceramic–composite resin interface can help protect against ceramic fracture and preserve the restoration's longevity.

Keywords*:* Adhesive remnant index; Hydrofluoric acid; Orthodontic bonding; Sandblasting; Shear bond strength; Silane; Surface roughness; Zirconium-based ceramics.

INTRODUCTION

Traditionally, orthodontic bonding involved fixing brackets to gold or stainless-steel bands, which posed challenges such as prolonged chairside time, patient discomfort, soft tissue irritation, plaque accumulation, and enamel damage.[1] However, with Buonocore's acid-etching technique in 1955 and further innovations by Newman, direct bonding has become widely adopted in clinical practice.[2-4] With the growing popularity of adult orthodontics and aesthetic dentistry, clinicians frequently need to bond orthodontic brackets to various restorative materials as well as enamel.[5] Previous studies have evaluated bracket bond strength to various substrates, including ceramic surfaces, temporary polycarbonate crowns, and aesthetic pontic materials such as porcelain.[6-8] Ceramics are favoured for repairing teeth due to their superior aesthetics, mechanical properties, and tissue compatibility. Commonly used in veneers, crowns, and bridges.[9] Zirconium is a significant component in ceramic restorations, serving as the foundation for all-ceramic crowns either through layered porcelain application or as monolithic crowns, prized for their strength and aesthetics achieved by overlaying with feldspathic porcelain.[10]

Despite advancements in direct bonding to surfaces beyond enamel, improving adhesive strength to composite or ceramic restorations while minimising bond failure remains a challenge. Chemical treatments like phosphoric acid and hydrofluoric acid (HFA), and mechanical methods such as diamond bur or sandblasting have been explored.[5] Standard mechanical enamel etching can enhance bond strength but risks damaging the restoration's glaze and ceramic integrity, leading to potential cracks and damage upon debonding.[11] Therefore, employing a blend of chemical treatments involving acids on the ceramic surface followed by silane application can preserve ceramic glaze while enhancing resin-ceramic adhesion.[11] Notably, there are few studies assessing orthodontic brackets' surface bond strength (SBS) on layered zirconia crowns. Hence, it is essential to establish dependable methods for adhering orthodontic brackets to layered zirconia restorations to guarantee sufficient strength. Hence, this research aims to assess the effects of three surface preparation protocols—diamond burs, sandblasting, and 9.6% HFA—on bonding metal orthodontic brackets to layered zirconia and evaluate their SBS. The study also examined how surface roughening influenced SBS and evaluated adhesive failure and ceramic integrity after debonding using the Porcelain Fracture Index (PFI) and Adhesive Remnant Index (ARI).

MATERIALS AND METHODS

The Experiment was conducted in the Department of Orthodontics and Dentofacial orthopaedics, SRM Dental College, Chennai with ethical clearance granted by the Institutional Review Board (SRMDC/IRB/2021/MDS/NO.101). Since the study did not involve human participants or patient data, informed consent was not required.

Fifty-two zirconia discs (8 mm diameter × 5 mm thickness) were designed using Autodesk 123D software and milled from Upcera Dental Zirconia Blank ST-D98-12 mm [Upcera Dental Technology Co., Ltd., China] via a CAD-CAM system (inLab CAM software, Dentsply Sirona, Germany). Following porcelain layering, each disc was mounted on a self-cure acrylic resin block (10 × 10 × 10 mm). The specimens were cleaned using compressed air free of oil after randomly distributed into four groups (n = 13 per group). There was no surface treatment given to Group I (control). Group II specimens were roughened using fine diamond burs [Mani CE-12F, Mani Inc., Japan] for 10 s. Group III underwent micro-etching with an intraoral sandblaster using 50 µm aluminium oxide particles (80 psi / 552 kPa) for 10 s. Group IV was treated with 9.6% hydrofluoric acid gel [Ultradent Products Inc., USA] for 60 s, then clean thoroughly with air–water spray. All specimens were dried with oil-free compressed air, coated with a silane primer [Ultradent Products Inc., USA] for 10 s, and light-cured.

Stainless steel pre-adjusted edgewise brackets for maxillary central incisors (Gemini series, 3M Unitek, Monrovia, CA, USA) were affixed using Transbond XT adhesive paste (3M Unitek, Monrovia, CA, USA) polymerised with light exposure for 20 seconds (Figure 2). After curing, samples were preserved in a water bath at 37 °C for 24 hours. After that, they were thermocycled 1,000 times, with a dwell time of 10 minutes between 5° and 55° Celsius.

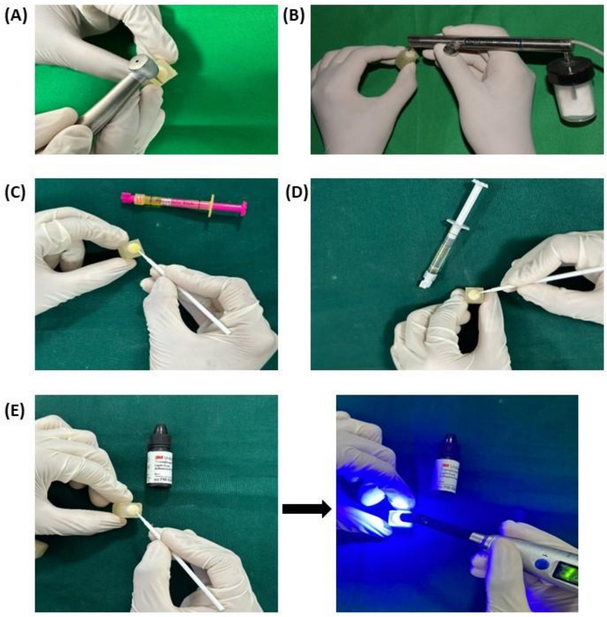

Figure 1. Surface treatment approached.
(A) Surface roughening with bur, (B) Sandblasting with intra oral sandblaster Al_2O_3, (C) Surface conditioning with 9.6% HFA, (D) Application of uniform layer of Silane coupling agent, (E) Application of thin layer of Transbond XT Primer followed by light curing.

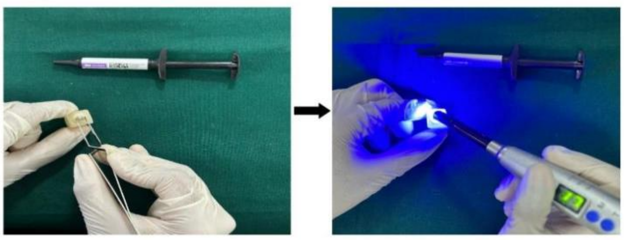

Figure 2. Bracket attachment and bonding using Transbond XT Adhesive followed by light curing.

2.1. Shear Bond Strength (SBS) Analysis

A universal testing machine (UTM) (Sensotech Technologies, India) was used to measure SBS. It operated with a crosshead speed precision of 0.01 mm/min and applied a load perpendicular to the specimen's long axis. A crosshead speed of 1 mm/min was used for the SBS test. The standard calculation formula was used to convert the force measured by the Instron unit, which was expressed in kilonewtons (kN), to megapascals (MPa).[1]

$$SBS\ (MPa) = \frac{Force\ (kN)}{Bonding\ area\ (mm^2)}$$

2.2. SEM Analysis for Surface Roughness Assessment

The samples were prepared by submerging them in a 2.5% glutaraldehyde solution for 12 hours, followed by cleaning with distilled water. After dehydration using a graded ethanol series, the specimens were left to air dry. To improve conductivity and avoid thermal damage, the specimens underwent chromium sputter-coating for scanning electron microscope (SEM) analysis. The morphology of the layered zirconia samples was then examined using an SEM (Thermoscientific Apreo S) under 1000x magnification to evaluate the surface harshness of the layered zirconia following each surface treatment.

2.3. Bracket Debonding Evaluation

The Adhesive Remnant Index (ARI) was used to identify the mode of bracket failure and to measure the amount of adhesive left after debonding, with scores ranging from 1 to 5 according to the method of Mehmeti et al. [12,13]

2.4. Evaluation of Ceramic Integrity

To evaluate the damage to the layered zirconia and determine the extent of the ceramic damage, the Porcelain Fracture Index (PFI) scoring system was employed.[14] According to the study by Mehmeti et al., the scores were categorised from 0 to 3.[15]

2.5. Statistical Analysis

Statistical analyses were carried out with IBM SPSS version 20.0. To summarise the SBS test results, descriptive statistics and a one-way ANOVA were used to compare the shear bond strength between groups. Bonferroni's method was employed for post-hoc testing to pinpoint particular variances between groups, with statistical significance determined by a p-value<0.001. Furthermore, a chi-square test (χ^2) was undertaken to examine the distribution of PFI and ARI scores among the four treatment groups, assessing whether the observed frequencies deviated significantly from the expected frequencies, considering categorical data.

3. RESULTS

3.1. Evaluation of Shear bond strength

The mean SBS across all groups was calculated to be 10.12 MPa, as depicted in Table 1. Notably, it was observed that sandblasting (Group III) yielded the highest mean SBS at 14.94 MPa, followed by treatment with 9.6% HFA (Group IV) at 13.94 MPa (Figure 3). Moreover, treatment with diamond burs (Group II) also demonstrated a higher mean SBS of 8.38 MPa compared to the control group (3.21 MPa). Each of the treatment groups exhibited improved bond strength, with Group III demonstrating the most favourable outcome.

Table 1. Descriptive Data for Shear Bond Strength

	N	Min (MPa)	Max (MPa)	Mean (MPa)	SD	SE	95% CI Lower	95% CI Upper
Group I (C) (Control)	13	1.54	5.20	3.21	1.28	0.36	2.43	3.98
Group II (B) (Diamond Burs)	13	6.30	10.50	8.38	1.38	0.38	7.54	9.21
Group III (S) (Sandblasting)	13	13.40	16.80	14.94	1.10	0.30	14.28	15.60
Group IV (H) (9.6% HFA)	13	10.90	17.60	13.95	2.23	0.62	12.60	15.30
Total	52	1.54	17.60	10.12	4.99	0.69	8.73	11.51

N: Number of participants; SD: Standard deviation; SE: Standard error; CI: Confidence interval.

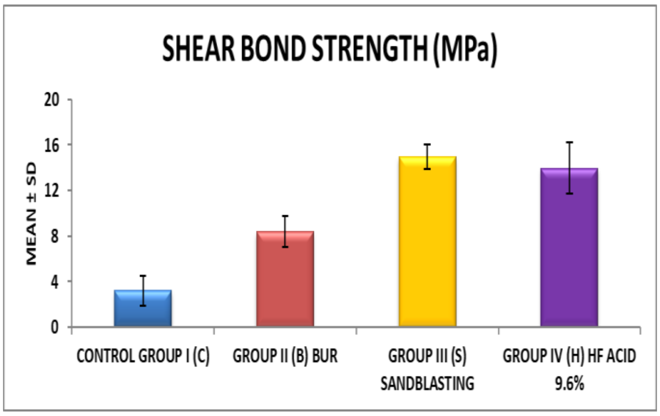

Figure 3. Assessing the shear bond strength across the groups.

3.2. SEM analysis

SEM images depicting the treated zirconia core surfaces are presented in Figure 4. Upon evaluation of the SEM images, minimal irregularity was observed in group II, treated with diamond burs (Figure 4(A)). Conversely, group III (sandblasting) (Figure 4(D)) and group IV (HFA) (Figure 4(C)) exhibited a significant level of irregularities, with group IV displaying the most effective surface roughening.

Analysis of variance (ANOVA) for SBS revealed statistically significant differences among the treatment groups. The variance in shear bond strength was primarily caused by differences between the treatment groups rather than within them, as indicated by the significantly higher between-group sum of squares (1153.776) than the within-group sum of squares (116.447) (Table 2).

The comparison between the groups was carried out using Tukey's post-hoc test, as shown in Table 3. Significant differences was noticed between Group I and Group II (mean difference=5.17 MPa, $p<0.0001$), Group I and Group III (mean difference=11.74 MPa, $p<0.0001$), Group I and Group IV (mean difference=10.74 MPa, $p<0.0001$), Group II and Group III (mean difference=6.57 MPa, $p<0.0001$), and Group II and Group IV (mean difference=5.57 MPa, $p<0.0001$). However, no significant difference was noted among the Group II and Group IV (mean difference=0.99 MPa, $p=0.664$).

Table 2. ANOVA testing for Shear bond strength

	Sum of Squares	df	Mean Square	F-value	P-value
Between Groups	**1153.776**	**3**	**384.592**	**158.531**	**<0.0001***
Within Groups	**116.447**	**48**	**2.426**		
Total	**1270.222**	**51**			

***p-value<0.05 is considered statistically significant. df: degrees of freedom.**

Table 3. Post Hoc Data for Shear Bond Strength

Group (I)	Group (J)	Mean Difference	SE	P-value
Group I (C) (Control)	**Group II (B) (Diamond Burs)**	**5.17***	**0.61**	**<0.0001***
	Group III (S) (Sandblasting)	**11.74***	**0.61**	**<0.0001***
	Group IV (H) (9.6% HFA)	**10.74***	**0.61**	**<0.0001***
Group II (B) (Diamond Burs)	**Group III (S) (Sandblasting)**	**6.57***	**0.61**	**<0.0001***
	Group IV (H) (9.6% HFA)	**5.57***	**0.61**	**<0.0001***
Group III (S) (Sandblasting)	**Group IV (H) (9.6% HFA)**	**0.99**	**0.61**	**0.664**

***p-value<0.05 is considered statistically significant. SE: Standard error.**

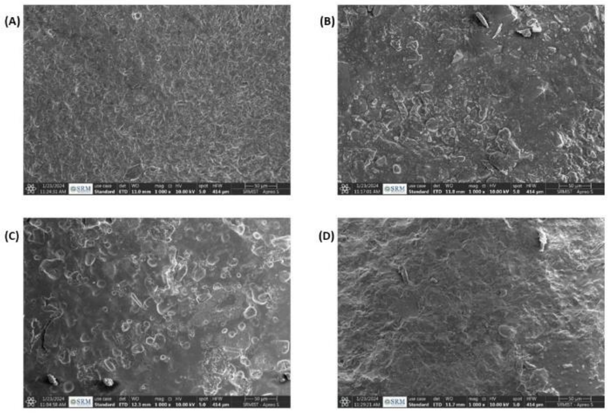

Figure 4. SEM images under 1000x magnification**.**

(A) Bur group, (B) Control group, (C) HFA group, (D) Silane group

3.3.1. Analysis of Adhesive remnant index

A chi-square test was employed to examine how the ARI was distributed across four treatment categories. The findings showed a noteworthy distinction in ARI among the groups ($\chi^2=41.795$, $p<0.001$). Significantly, ARI proportions were higher in Group III (Sandblasting) and Group IV (HF Acid 9.6%) than in the control and bur treatment groups (Table 4). The proportionate spread of ARI scores between the four groups exhibited a remarkably significant statistical contrast ($p < 0.0001$).

Table 4. Statistical data for Adhesive remnant index

ARI Index	Group I (C)	Group II (B)	Group III (S)	Group IV (H)	Total	χ2	P-value
All adhesive remaining on the ceramic crown surface with the impression of the bracket base	0 (0%)	0 (0%)	2 (15.4%)	2 (15.4%)	4 (7.7%)	41.795	<0.0001*
More than 90% of the adhesive remaining on the ceramic crown surface	0 (0%)	0 (0%)	2 (15.4%)	5 (38.5%)	7 (13.5%)		
Less than 90%, but more than 10% of the adhesive remaining on the surface	0 (0%)	2 (15.4%)	6 (46.2%)	2 (15.4%)	10 (19.2%)		
Less than 10% of the adhesive remaining on the ceramic crown surface	2 (15.4%)	4 (30.8%)	3 (23.1%)	4 (0.8%)	13 (25%)		
No adhesive remaining on the ceramic crown surface	11 (84.6%)	7 (53.8%)	0 (0%)	0 (0%)	18 (34.6%)		

***p-value<0.05 is considered statistically significant.**

3.3.2. Analysis of Porcelain fracture index

The chi-square test was employed to evaluate the distribution of Porcelain Fracture Index (PFI) within the four treatment categories. The results showed a marked variation in PFI scores among the experimental sets (χ^2=20.233, p=0.003), indicating considerable differences in porcelain fracture outcomes between the treatments. Group I (C) showed no instances of surface damage, while Group II (Bur), Group III (Sandblasting), and Group IV (HF Acid 9.6%) exhibited higher rates of surface damage in relation to the control group (Table 5).

Table 5. Statistical data for Porcelain Fracture index

PFI	Group I (C)	Group II (B)	Group III (S)	Group IV (H)	Total	χ2	P-value
ceramic surface intact or in the same condition as before the bonding procedure	11 (84.6%)	2 (15.4%)	2 (15.4%)	3 (23.1%)	18 (34.6%)	20.233	0.003*
surface damage limited to glaze layer or very superficial ceramic	2 (15.4%)	9 (69.2%)	9 (69.2%)	7 (53.8%)	27 (51.9%)		
surface damage, which features a significant loss of ceramic requiring restoration of the defect	0 (0%)	2 (15.4%)	2 (15.4%)	3 (23.1%)	7 (13.5%)		

*p-value<0.05 is considered statistically significant.

4.0. DISCUSSION

In the current study, silane was applied after either mechanical or chemical surface conditioning to enhance SBS. This combination produced a significant improvement in bond strength, consistent with the observations reported by Wood et al. (1986) and Schmage et al. (2003).[16,17] A mean SBS of 3.20MPa in the control group indicates that the silane used has high water solubility, implying that using silane alone may not be sufficient. Therefore, it should be incorporated with either mechanical or chemical surface roughening. This finding aligns with the research conducted by PD Vaishnav et al. (2015), who also noted similar outcomes in the control group of their study.[18] Both sandblasting and the 9.6% HFA treated group showed enhanced SBS with a negligible difference; however, sandblasting gave a more effective SBS compared to 9.6% HFA. Our study shows similar findings to the work of Schmage et al. (2003), where treatment with sandblasting was more effective compared to chemical etching with HFA.[17] Further, sandblasting with AL_2O_3 treated group showed higher bond strength compared to roughening with diamond burs. A comparable association was observed when brackets were bonded to metal restorations, where mechanical retention showed improvement.[19]

Silane application plays a key role in promoting adhesion between ceramics and composite resins. Nevertheless, the possible weakening effect of thermocycling on silane bonds, particularly after hydrofluoric acid etching.[17] Mechanical roughening through diamond burs and sandblasting, excluding the use of ceramics, induces crack formation, which typically persists in the mouth after debonding. Hence, it's imperative to prevent any harm to the ceramic surface.[18] SEM results show that using diamond burs leads to smoother surfaces with fewer irregularities, while sandblasting or HFA treatment is more effective at roughening the surface. On the contrary, treatment with sandblasting or HFA results in a most effective surface roughening. The ARI was assessed to classify the bracket bond failure type and quantify the residual material left on the layer after bracket removal.[20] The ARI scores showed statistical significance ($p<0.0001$), with each adhesive staying intact on the surface of the ceramic crown, leaving a mark on the bracket base in both group III and group IV at a rate of 15.4%. However, no adhesive residue was observed on the ceramic crown surface in 84.6% of the control group and 53.8% of Group II. The bonding detachment at the ceramic–composite interface was seen in the majority of specimens, suggesting that the cohesive strength of the composite and the bracket–composite bond were both higher than the bond between the composite and the ceramic surface.

Ideally, it is preferred that adhesive failures happen at the junction between the composite and ceramic to avoid ceramic cracks when removing the bond.[19] Cohesive failures within ceramics typically occur when the bond strengths among ceramic and composite resin surpass 13 MPa.[21] In the current study, the majority of groups recorded SBS values above 13 MPa, with adhesive failures observed but no evidence of ceramic cracks or fractures. The findings align with the study by Harari et al. (2003), which also reported bonding failure in the HFA and APA groups. From a clinical standpoint, the absence of detectable ceramic damage suggests improved preservation and long-term stability of the restoration.[22]

The PFI was employed to evaluate any impairments on the layered zirconia surface, chi-square test comparing between the four group's index scores, revealing statistically significant variances ($p < 0.0001$). In the control group, 84.6% of specimens showed no alteration of the ceramic surface compared to its pre-bonding condition, whereas this proportion was only 15.4% in groups II and III, and 23.1% in group IV. Minor surface changes confined to the glaze layer or the superficial ceramic were recorded in 15.4% of the control group, increasing to 69.2% in groups II and III and 53.8% in group IV. More severe ceramic loss, necessitating restoration, occurred in 15.4% of specimens from groups II and III and in 23.1% of group IV, while no such cases were noted in the control group. These outcomes are aligned with the results of Alhaija et al. (2010).[8] This indicates that hydrofluoric acid treatment may enhance the vulnerability of ceramic materials in general. However, as this is an in vitro study, the clinical correlation may vary due to factors like masticatory force and oral environment changes, warranting further clinical investigations. Future studies should also address the chemical impact of 9.6% HFA application on patient crowns.

5.0. LIMITATION OF THE STUDY

Despite the fact that this study offers insightful information, some limitations must be acknowledged. Since the experiment was conducted under laboratory conditions, the outcomes may not fully reflect the dynamic oral environment, where factors such as saliva, chewing forces, and pH variations continuously affect bonding. Although sufficient for initial assessment, the sample size restricts the extent to which the findings can be applied. Furthermore, the testing was limited to three surface conditioning techniques; laser treatment, tribochemical silica coating, and plasma applications were not included. Another point to consider is that the long-term effects of repeated ageing and thermal cycling were not evaluated, which may influence bond stability over time. Future research should concentrate on clinical trials that include a wider range of surface treatments, longer follow-up times, and larger populations. Such studies would guarantee the durability of zirconia restorations in practical environments and aid in the development of more consistent bonding protocols.

6.0. CONCLUSION

The study demonstrated that sandblasting and chemical conditioning with 9.6% HFA are effective methods for enhancing the bond strength of orthodontic procedures. However, these treatments may also result in higher surface damage, so it is essential to select appropriate bonding techniques and materials to minimise the risk of porcelain fracture and preserve ceramic surface integrity. Nevertheless, clinical validation is needed before these approaches can be routinely recommended

DECLARATIONS

Conflicts Of Interests

None

Funding

None

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