Effect of Different Surface Treatments on Shear Bond Strength between Zirconia and Metal Orthodontic Brackets

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Abstract

The chemical inertness and uniform texture of zirconia make it difficult to establish a strong bond between orthodontic brackets and zirconia surfaces. This study evaluated the effect of different mechanical surface treatments on the shear bond strength (SBS) between orthodontic brackets and zirconia. Sixty zirconia specimens (8 × 8 × 2 mm) were divided into six groups according to surface treatments: no surface treatment (C), sandblast with 50 µm Al_2O_3 (SB), diamond bur grinding with extra fine 15 µm (DE), fine 40 µm (DF), medium 80 µm (DM), and coarse 120 µm (DC) grit sizes. A contact profilometer measured surface roughness (Ra). All specimens were treated with Clearfil Ceramic Primer Plus, and Transbond XT adhesive was bonded with orthodontic brackets. SBS testing was performed following thermocycling. Stereomicroscopy was used to investigate failure modes; the adhesive residue was measured with the Adhesive Remnant Index (ARI). Statistical analysis was performed using one-way ANOVA ($p < 0.05$). Group DE (15 µm) demonstrated the highest SBS (14.56 ± 4.02 MPa), significantly higher than Groups DM and C ($p < 0.05$). SBS values of all groups exceeded the minimum clinical threshold (5.89–7.85 MPa), indicating their clinical applicability. Most specimens showed an ARI score of 1, suggesting that more than half of the adhesive remained on the bracket base.

In conclusion, mechanical treatment with extra-fine diamond bur grinding, followed by chemical conditioning with a 10-MDP-containing primer, significantly improves bracket bonding to zirconia. This protocol may offer a reliable and effective method for orthodontic bracket adhesion in patients with zirconia restorations.

Keywords: *shear bond strength, mechanical surface treatment, zirconia, metal bracket and orthodontic bracket*

1. Introduction

Orthodontics is a dental specialty that focuses on identifying, preventing, and treating malocclusions. Fixed orthodontic appliances, also known as braces, focus on treating dental and skeletal problems. Brackets and archwires attached to teeth provide precise, consistent forces for effective tooth movement (Proffit et al., 2018). Due to the excellent characteristics of zirconia, sometimes known as zirconium oxide (ZrO_2), the substance has become popular in dentistry. The zirconia has excellent mechanical strength, biocompatibility, esthetics, and resistance to wear and chemical degradation. These features make zirconia appropriate for dental restorations, implants, and orthodontic treatment (Parthasarathy et al., 2024; Piconi, & Maccauro, 1999).

The special properties of zirconia produce particular challenges for obtaining a strong bond. Zirconia is chemically inert and stable and does not easily bond with conventional dental adhesives. Many dental ceramics include silica, which helps them to adhere effectively following hydrofluoric acid treatment. However, zirconia lacks silica; hence, hydrofluoric acid etching is not suitable for achieving a strong adhesive bond (Nejat, 2024). This makes conventional bonding methods inadequate for surfaces of zirconia. Additionally, smooth surfaces of zirconia decrease the surface area accessible for adhesives to bond. While surface roughness is commonly used in adhesives to produce mechanical retention, zirconia's natural smoothness makes this challenging (Fischer, 2024).

Many previous studies had proposed methods to enhance bonding to zirconia in order to overcome its bonding difficulties, mechanical surface treatments by using sandblasting. This method produces a rough surface that increases the bonding area availability. Studies revealed that although sandblasting greatly increases bond strength but, mechanical surface treatment may weaken the zirconia by generating micro-cracks (Inokoshi et al., 2021; Menees et al., 2014; Zhang et al., 2004). Surface grinding by using diamond burs to grind zirconia creates grooves that allow adhesives to bond better. But, if done improperly, this technique can weaken zirconia, much as sandblasting (Lee et al., 2019; Lundberg et al., 2017).

Chemical surface treatments applying primers comprising 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). These primers build a chemical contact with zirconia and generate a strong zirconium-phosphate layer that improves the binding strength between zirconia and adhesives. Usually, the combination of mechanical and chemical treatments yields better outcomes. For instance, stronger bindings are produced by sandblasting followed by a 10-MDP primer than by utilizing either technique alone (Nagaoka et al., 2017; Yue et al., 2019).

In orthodontics, bracket bonding is important for treatment success. Brackets must remain connected to the tooth/crown despite continual forces applied during tooth movement, which can put a strain on the adhesive bond and may cause bond failure during orthodontic tooth movement. Bond failures delay treatment progress, extend treatment duration, and need additional dental visits (Denry, & Kelly, 2008). Achieving optimal shear bond strength (SBS) between orthodontic brackets and tooth or restorative surfaces influences treatment efficiency and success in orthodontic treatment. A weak bond can cause brackets to become dislodged when orthodontic force or masticatory pressures occur, consequently interrupting treatment and extending the length of treatment, requiring more chairside adjustments, and making patients more uncomfortable (Bishara et al., 2005; Cal-Neto et al., 2006). This problem has become more relevant in adult orthodontics, where bonding to restorative materials such as zirconia is somewhat widely used. Given zirconia's extreme hardness and chemical inertness, conventional bonding methods may be ineffective; so, new efficient bonding procedures are needed to obtain a safe and strong bracket bond. The bond should also be strong enough to resist clinical standards but safe enough to be debonded at the end of treatment without affecting the zirconia restoration (Seo, 2014; Kim et al., 2017). Thus, research aiming to improve SBS to zirconia is essential in modern orthodontic treatment to guarantee predictable results and prevent future problems.

2. Objectives

Prior studies have examined the shear bond strength between mechanically treated zirconia surfaces and metal brackets. It is widely recognized that 10-MDP facilitates the bonding of zirconia to metal brackets. It has been demonstrated that 10-MDP facilitates the bonding of zirconia to metal brackets. However, the mechanism by which it combined mechanical surface treatments with various grit sizes of diamond burs and applied 10-MDP may not be widely understood. This study aimed to examine the effect of various mechanical surface treatments of zirconia on the shear bond strength (SBS) between orthodontic brackets and zirconia.

Null hypothesis: The shear bond strengths between zirconia and metal brackets were not different at various surface-conditioning levels: aluminum oxide sandblasting (50 µm), diamond bur grinding extra fine (15 µm), fine (40 µm), medium (80 µm), and coarse (120 µm).

Alternative hypothesis: The shear bond strengths between zirconia and metal brackets were different at various surface-conditioning levels: aluminum oxide sandblasting (50 µm), diamond bur grinding extra fine (15 µm), fine (40 µm), medium (80 µm), and coarse (120 µm).

3. Materials and Methods

The sample size was determined by using G*Power 3.1 Software (University of Dusseldorf, Germany) and setting the significance level at 0.05, the power at 0.99, and the effect size at 0.947 based on the results of a previous study by Amer, and Rayyan (2018). The results indicated that a sample size of 8 specimens per group was required for this study. However, in this study, a sample size of 10 per group (total sample size of 60 specimens) was considered appropriate.

Table 1 materials adopted in the present study, their compositions and manufacturers

Materials	Composition	Manufacturers
Pre-sintered zirconia blocks VITA YZ HT	Zirconium oxide (ZrO_2) = 90- 95% Yttrium oxide (Y_2O_3) = 4-6. Hafnium oxide (HfO_2) = 1-3% Aluminum oxide (Al_2O_3) = 0-1%	Vita Zahnfabrik, H. Rauter GmbH & Co. KG, Postfach, Bad Säckingen, Germany
Clearfil Ceramic Primer Plus	Ethanol > 80% 3-Trimethoxysilylpropyl methacrylate 10-MDP	Kuraray Noritake Dental Inc., Nagoya, Japan
Transbond XT adhesive	Silane Treated Quartz = 70-80% Bis-GMA = 10-20% BISEMA = 5-10% Silane Treated Silica < 2 % Diphenyliodonium Hexafluorophosphate < 0.2 %	3M Unitek, Monorvia, CA, USA
Sandblasting powder	Aluminum oxide (Al_2O_3) 50µm ≥ 99.5% Silicon dioxide (SiO_2) < 0.6%	Renfert cobra Untere Giesswiesen 2, Hilzingen, Germany
Diamond bur (15, 40, 80, 120µm)	Diamond cylinder round edge (Length = 10 mm, diameter = 1.4 µm)	Via al Molino, Montagnola, Switzerland

According to the information provided by the manufacturers:
Bis-GMA: bisphenol-A-diglycidylmethacrylate;
BISEMA: Bisphenol A Bis (2-Hydroxyethyl Ether) Dimethacrylate;
10-MDP: 10-methacryloxydecyl dihydrogen phosphate
µm: microns

3.1 Materials Preparation

Sixty pre-sintered Y-TZP zirconia blocks were cut into pieces measuring 10 mm height, 10 mm width, and 2 mm thickness were prepared from a zirconia block (Vita Zahnfabrik, H. Rauter GmbH & Co. KG, Postfach, Bad Säckingen, Germany) using a cutting machine (Accutom-50, Struers. Inc, Cleveland, USA). The cut pieces were then polished with a polishing machine with 800 grit silicon carbide paper (3M Wetordry abrasive sheet, 3M Minnesota, USA) with 100 rounds/minute for 2 minutes under running water using an automatic polishing machine (Tegramn-25, Struers. Inc, Cleveland, USA) After polishing; the specimens were sintered following the manufacturer's instructions.

3.2 Experimental Groups

The mechanical surface treatments were categorized into three major groups.

The specimens were randomly assigned to six groups based on surface treatment:

1. **Control Group C (Control):** No surface treatment. The zirconia surface was left untreated, serving as the control group.

2. **Sandblast Group SB (Sandblasting):** Sandblasted with 50 µm aluminum oxide. The zirconia surface was sandblasted with 50 µm aluminum oxide particles at a pressure of 0.2 MPa for 20 seconds, maintaining a distance of 10 mm. The sandblasting procedure used a sandblasting unit (Microetcher II intraoral sandblaster; Danville materials, SAN RAMON, CA, USA). The direction of the sandblaster was precisely controlled using a Computer Numerical Control (CNC) lathe. During operation, the specimen holder of the CNC lathe moved while the sandblaster remained stationary.

3. **Diamond bur Group DE, DF, DM, DC (Extra fine, Fine, Medium, Coarse):** Diamond bur grinding with different grit size. The zirconia surface was ground with 15 µm (DE), 40 µm (DF), 80 µm (DM), and120 µm (DC) grit-size diamond bur, respectively. The zirconia surface was mechanically treated using a standard cylindrical diamond bur with a different grit size (Intensiv SA, Via al Molino, Montagnola,

Switzerland) on a high-speed dental handpiece. The direction of the bur was precisely controlled using a Computer Numerical Control (CNC) lathe. The flat surface of the diamond bur was aligned parallel to and in contact with the flat surface of the zirconia specimen. During operation, the specimen holder of the CNC lathe moved in a single straight direction from left to right while the diamond bur remained stationary.

3.3 Surface Roughness (Ra) Measurement

The average surface roughness value (Ra) of all specimens was measured by using a contact profilometer TalyScan 150 (Taylor Hobson, Leicester, England) and associated software. Measurement was recorded to evaluate the average surface roughness changes resulting from the mechanical surface preparation of zirconia surfaces. The profilometer was set, and the stylus tip was moved across the surface with a travel speed of 1 mm/s. Five readings were recorded for each zirconia specimen, each reading 0.8 mm apart, and then the average Ra was calculated.

3.4 Bonding Procedure

The specimens were embedded in polyvinyl chloride (PVC) rigid rings and filled with dental acrylic to create stable holders for testing. The zirconia surface was applied with 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) containing a bonding agent Clearfil Ceramic Primer Plus (Kuraray Noritake Dental Inc., Nagoya, Japan). The metal brackets (Unitek Gemini Brackets MBT; 3M Unitek, Monrovia, CA, USA) were bonded onto the zirconia surface with a light-cured adhesive resin (Transbond XT, 3M Unitek, Monorvia, CA, USA). The excess adhesive was removed with a fine explorer. The adhesive was light cured using a VALO® Ortho curing light (Ultradent Products, Inc., South Jordan, UT, USA) for 3 seconds on mesial and distal sides (Ward et al., 2015). All specimens were then artificial aging with a thermocycling machine (Thermocycler, SD mechatronik, Munich, Germany). Thermocycled in distilled water between 5°C and 55°C for 10000 cycles the immersion time in each bath was 30 s, and the dwell time was 10 s (Gale, & Darvell, 1999).

3.5 Shear Bond Strength Testing

The bracket area of each specimen was measured before the analysis of the shear bond strength. A universal testing machine tested The samples for shear bond strength (AGS-X 500N, Shimadzu Corporation, Kyoto, Japan). Each specimen was fixed in the testing machine, and the shearing blade was placed parallel to the junction between the zirconia and resin cement, as shown in Figure 1. The shear load was applied at a 0.5 mm/min crosshead speed. The shear bond strength (MPa) was calculated by dividing the highest shear bond strength by the surface area of the zirconia-bracket interface.

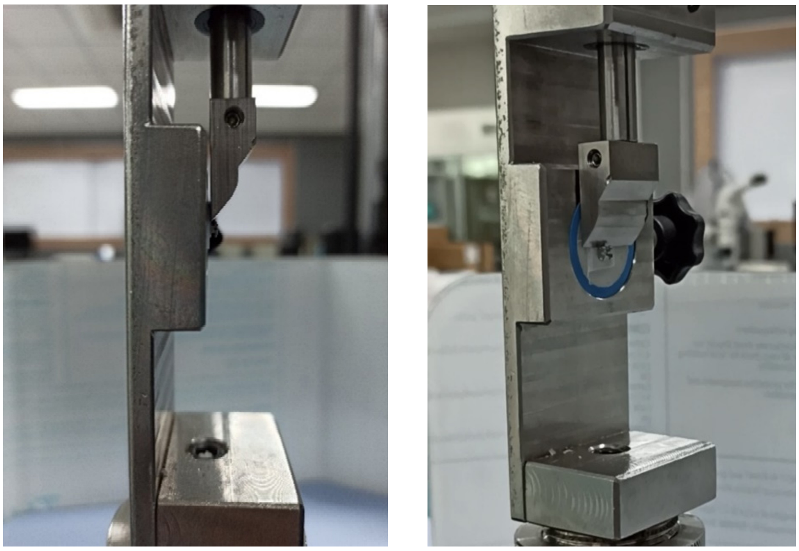

Figure 1 Specimen was placed in the testing jig with the universal testing machine

3.6 Mode of Failure Analysis

After shear bond strength testing, all bracket surfaces of each specimen were examined under a stereomicroscope at a magnification of ×10 as shown in Figures 2-3 to evaluate and using Image J software (ImageJ® software, Maryland, USA) for assessment and the Adhesive Remnant Index (ARI) was calculated based on the following modified scale as adapted from Årtun, and Bergland (1984) which was evaluated the amount of adhesive left on the bracket surface;

score 0 = the entire adhesive left on the bracket base,
score 1 = more than half of the adhesive left on the bracket base,
score 2 = less than half of the adhesive left on the bracket base,
score 3 = no adhesive left on the bracket base.

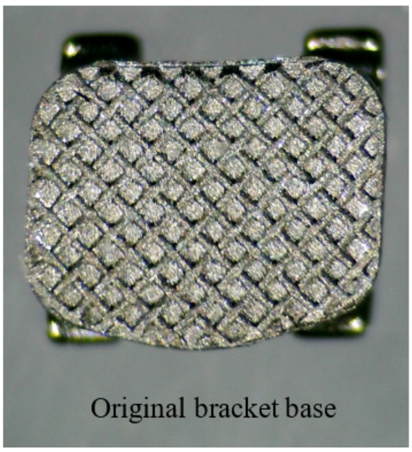

Figure 2 Illustration from stereomicroscope image of bracket base before bonding, showing surface condition for ARI comparison.

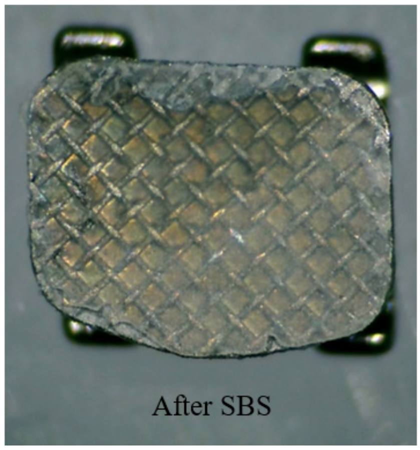

Figure 3 Illustration from stereomicroscope image demonstrating ARI score 1, indicating more than half of the adhesive remained on the bracket base after debonding.

The examples of stereomicroscopic images of the bracket base before bonding are presented in Figure 2, and stereomicroscope images of specimens with the mode of failure score one are presented in Figure 3.

3.7 Statistical Analysis

The results of surface roughness and shear bond strength of all groups were analyzed using SPSS 20.0 software software (SPSS Inc, Chicago, Illinois, United States), setting the confidence level at 95%. The normality of distribution was tested by the Kolmogorov-Smirnov test (KS test) and the homogeneity of variance was tested using Levene's test. The bond strength values were further analyzed using one-way ANOVA to assess the primary outcome and followed by post hoc Tukey's test to assess the multiple comparisons (Table 2).

Mode of failure: The Chi-square test was used to evaluate the scores among the adhesive subgroups. Statistical significance will be set at $P<0.05$, considered as being statistically significant.

Pearson's correlation analysis was conducted to assess the relationship between surface roughness (Ra) and shear bond strength (SBS).

4. Results and Discussion

4.1 Results

4.1.1 Zirconia Surface Roughness

The multiple comparisons test revealed that The highest surface roughness was observed in group DC (0.123 ± 0.007MPa), followed by group DM (0.117 ± 0.013 MPa), group DF (0.105 ± 0.008 MPa), group DE (0.063 ± 0.028MPa), group SB (0.058 ± 0.012MPa), and the smoothest surface was group C (0.048 ± 0.022 MPa) (Table 2). The surface roughness of group DF, DM, and DC was significantly higher than that of group C, SB and DE ($p < 0.05$). However, there were no significant differences between groups DF, DM, and DC. Additionally, there were also no significant differences between groups C, SB and DE

Table 2 Descriptive Statistics of Zirconia Roughness (N = 60)

Group	Mean (µm)	SD (µm)	Min (µm)	Max (µm)
Group C (no treatment)	0.048	0.022	0.033	0.103
Group SB (sandblast 50 Microns)	0.058	0.012	0.042	0.084
Group DE (diamond bur 15 Microns)	0.063	0.028	0.016	0.094
Group DF (diamond bur 40 Microns)	0.105	0.008	0.092	0.118
Group DM (diamond bur 80 Microns)	0.117	0.013	0.103	0.141
Group DC (diamond bur 120 Microns)	0.123	0.007	0.113	0.136

4.1.2 Shear Bond Strength Test

The shear bond strength (SBS) results demonstrated that Group DE (diamond bur 15 µm) obtained the highest SBS (14.56 ± 4.02 MPa), followed by Group DF (12.73 ± 3.78 MPa), Group DC (12.73 ± 3.30 MPa), Group SB (10.12 ± 3.88 MPa), Group DM (8.96 ± 3.81 MPa), and Group C (8.26 ± 1.36 MPa). Group E exhibited a statistically significant higher SBS in comparison to Groups DM and C ($p < 0.05$); however, no significant differences were detected among Groups C, SB, DF, DM, and DC. Table 3 summarizes the SBS results and depicts the distribution of failure modes according to the Adhesive Remnant Index (ARI).

4.1.3 Mode of Failure Analysis

The percentage distribution of Adhesive Remnant Index (ARI) scores for each group is presented in Table 3. Notably, only scores of 0 and 1 were observed across all groups. Statistical analysis demonstrated that the score distribution did not differ significantly among the groups ($p < 0.05$).

4.1.4 Relationship between Surface Roughness (Ra) and Shear Bond Strength (SBS).

A Pearson's correlation analysis was conducted to assess the relationship between surface roughness (Ra) and shear bond strength (SBS). The analysis revealed a weak positive correlation ($r = 0.198$, $p = 0.707$), indicating no statistically significant relationship between Ra and SBS among the six groups.

Table 3 Mean shear bond strengths and percentages of failure mode

Groups	Mean shearbond strength (SD)	Mode of failure			
		Score 0	Score 1	Score 2	Score 3
Group C (no treatment)	8.259 (1.36)	0	100	0	0
Group SB (sandblast 50 Microns)	10.123 (3.88)	0	100	0	0
Group DE (diamond bur 15 Microns)	14.562 (4.02)	20	80	0	0
Group DF (diamond bur 40 Microns)	12.728 (3.78)	0	100	0	0
Group DM (diamond bur 80 Microns)	8.955 (3.81)	0	100	0	0
Group DC (diamond bur 120 Microns)	12.727 (3.30)	0	100	0	0

4.2 Discussion

The present study investigated the effect of different mechanical surface treatments followed by chemical surface treatment on the shear bond strength between zirconia and metal brackets. The results showed that the larger the instrument for grinding/sandblasting, the higher the value of the surface roughness (Ra); Group DC (diamond bur 120 µm), DM (diamond bur 80 µm), and DF (diamond bur 40 µm) were significantly higher than Group DE (diamond bur 15 µm), SB (sandblast 50 µm), and C (no surface treatment), while there was no significant difference between Group DF, DM, DC and also no significant difference between Group C, SB, DE. Therefore, the null hypothesis was rejected and the alternative hypothesis was accepted.

The means of SBS from this study presented that all groups, including the no surface treatment group, had SBS values more than the minimum bond strength of 5.89-7.85 MPa (Reynolds, 1975), and another study 6-8 MPa (Bourke, & Rock, 1999) which was reasonable for clinical use. Even group control had no mechanical surface treatment but still had means SBS more than minimum bond strength due to chemical surface treatment with bonding contained 10-MDP in which one P-OH non-deprotonated of the phosphate group from 10-MDP strongly interacts with zirconia's oxide layer, forming a stable nano-layered structure zirconium-phosphate bonds. These chemical interactions enhance adhesive penetration and create long-term stability, preventing bond degradation over time (Nagaoka et al., 2017; Yoshida et al., 2005).

This study used a 10-MDP-based primer and several bonding techniques, including universal adhesives, for the bonding of metal brackets to zirconia. Pouyanfar et al., (2019) examined the efficacy of Scotchbond Universal (3M), a universal adhesive containing MDP, applying several surface treatments such as sandblasting, acid etching, and untreated surfaces. Their findings indicated that both the untreated (12.85 ± 7.16 MPa) and acid-etched (9.89 ± 9.28 MPa) groups had clinically acceptable bond strengths, but sandblasting provided the highest shear bond strength (19.25 ± 9.07 MPa). However, there was no statistically significant difference between the groups ($p = 0.06$) despite SBS values differences. The results correspond with our research, which also showed that all groups, including the control, achieved the minimum clinical SBS standards. This emphasizes the efficacy of MDP-containing bonding agents, whether used with specific zirconia primers or universal adhesives. Moreover, research shows that surface preparation can increase bond strength, yet, in the case of chemically active adhesives, it may not be necessary.

The extra-fine diamond bur group (15 µm) had the highest shear bond strength (SBS), even though its surface was smoother than the coarse, medium, and fine diamond bur groups. This discovery suggests that surface roughness alone may not be the primary determinant of bond strength. This finding corresponds with Pearson's correlation analysis results, which demonstrated no association between surface roughness (Ra) and SBS ($r = 0.198$, $p = 0.707$), suggesting no statistically significant relationship. The lack of association also supports the idea that an increase in surface roughness does not necessarily result in enhanced bond strength. One possible explanation is that excessively rough zirconia surfaces may not establish optimal contact with the substrate because of their irregular topology, which would make it more difficult for the resin to be distributed properly. Smoother surface profiles, such as those created by extra-fine diamond burs, may enhance adhesive adhesion and interaction at the microscopic level.

This study did not specifically evaluate surface morphology or phase transformation, but previous investigations have reported similar results. Researchers De Araújo Michida et al., (2015) found that particular aggressive grinding methods could compromise zirconia's structural integrity. Mosharraf et al., (2011) indicated that excessively rough surfaces may lead to microcrack development, potentially undermining long-term bonding durability. The precise mechanisms underlying these outcomes remain complex and involve several variables. Further investigation into the microstructural consequences of surface treatments is necessary, employing analytical techniques such as Scanning Electron Microscopy (SEM) or X-ray Diffraction (XRD).

Based on these results, the current study does not recommend the use of diamond bur that has a grit size of more than 40 microns. The enhanced SBS observed in the extra-fine diamond bur group could be attributed to factors such as improved surface area, optimized adhesive penetration, or modifications in the microstructural characteristics of the prepared zirconia. These results show that more research is needed to fully understand how the underlying mechanisms affect the bond strength outcomes in different surface preparation methods.

Most of the specimens mainly had an ARI score of 1, which is an indicator of bond failure between the zirconia surface and the adhesive. Considering that the greater amount of adhesive remaining on the bracket surface means less chance of zirconia damage and also easier and safer for debonding the bracket.

Within the limitation of the present study, from the result, it could be applied in the clinical situation when the zirconia surface needs to bond with an orthodontic metal bracket; the extra fine diamond bur grinding (15 microns) followed by 10-MDP bonding provides a better bond strength. However, the clinician should be aware that too much mechanical surface treatment may cause phase transformation of zirconia and compromise the SBS between zirconia and metal bracket. Further studies should be investigated on different methods for mechanical surface treatment of zirconia with different bonding agents.

The findings of this study provide significant insights into the effects of mechanical surface treatments and 10-MDP primer application on zirconia bonding, although certain limitations must be considered. The study did not assess the long-term endurance of bonds subjected to cyclic loading or artificial aging methods other than thermocycling. Resistance to fatigue and aging stability is essential for accurately simulating intraoral conditions; future investigations should integrate techniques such as cyclic mechanical loading or water storage over extended periods. Secondly, only one adhesive primer, 10-MDP-promer, was evaluated. While 10-MDP is extensively utilized, alternate bonding agents, like silane-based primers, GPDM-containing adhesives, or universal adhesives, may provide additional or alternative benefits and should be explored in future research. Lastly, as this was an in vitro experiment, the results may not entirely replicate the variable conditions of the oral environment. Therefore, clinical trials are essential for evaluating the efficacy and durability of the bonding technique in orthodontic treatment.

5. Conclusion

Mechanical surface treatment of zirconia by using the extra fine diamond bur grinding (15 microns) followed by chemical surface treatment by using 10-MDP bonding provides a better bond strength significantly. However, the clinician should be aware that too much mechanical surface treatment may cause phase transformation of zirconia and compromise the SBS between zirconia and metal bracket.

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