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Effect of Acid Surface Treatments on the Shear Bond Strength of Metal Bracket to Zirconia Ceramics

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Abstract: The surface treatment of zirconia prior to bonding remains controversial and unclear. This study aimed to compare the shear bond strength (SBS) of metal brackets to zirconia under surface treatments with either 4% HF or 37% PA in both immediate loading (IML) and artificial aging by thermocycling (TMC). Methods: Eighty-four zirconia were randomly assigned to six groups based on the surface treatment and artificial aging by TMC: (1) No surface treatment (NT); (2) NT + TMC; (3) HF (4% HF for 2 min); (4) HF + TMC; (5) PA (37% PA for 2 min); and (6) PA + TMC. After bracket bonding, only the TMC groups were thermocycled for 5000 cycles. The SBS and adhesive remnant index (ARI) of all groups were analyzed ($p < 0.01$). Results: TMC significantly lowered the SBS more than the IML in all acid surface treatment groups ($p < 0.01$). The ARI score after TMC was significantly higher than the IML in all acid surface treatment groups ($p < 0.001$). No significant differences in the SBS values or ARI scores were observed among the surface treatments ($p > 0.01$). Conclusions: Two-minute simple etching methods, using either 4% HF or 37% PA, showed an insufficient SBS of metal orthodontic brackets to zirconia after TMC.

Keywords: ceramic materials; hydrofluoric acid; metal brackets; phosphoric acid; shear bond strength

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1. Introduction

Aesthetic concerns among dental patients have shifted the paradigm from metal-based restorations towards all-ceramic restorations. Zirconia is commonly recognized for its superior mechanical qualities [1,2], biocompatibility, aesthetics, and easy digital fabrication through computer-aided design/computer-aided manufacturing (CAD/CAM) technology [3–5]. This suggests the greater probability of a dentist encountering zirconia restorations in the oral cavity during orthodontic treatment.

Due to the inert nature of zirconia, desirable bracket bonding to zirconia is very challenging for orthodontists [6]. Reynolds [7] and Thurmond et al. [8] reported an acceptable orthodontic bond strength to ceramic materials that ranged from 4.90 MPa to 13.00 MPa. The ideal bond strength in orthodontics should have sufficient strength to withstand the occlusal force but should not be too strong to break the ceramic surface during debonding [9]. Adequate resin–zirconia bonding can be achieved through mechanical and chemical surface treatment on zirconia materials prior to the bonding procedure [6,10–12].

Mechanical surface treatment aims to roughen the zirconia surface to allow the resin to establish a micromechanical interlock [6,13]. Previous literature have suggested several methods that included airborne-particle abrasion, silica coating, laser irradiation, and acid etching [6,10–12,14]. The first three techniques showed a stronger bond strength than the acceptable range, while the acid etching technique remains controversial [14–16].

Hydrofluoric acid (HF) from 4% to 10% is commonly used in ceramic surface treatment [17,18]. Previous studies showed that zirconia was resistant to acid etching due to minimal silica content [3,6,10]. Although HF-etched zirconia resulted in a low shear bond strength (SBS) and minimal surface topographic changes [17], the SBS was within the acceptable range for bracket bonding [19–21].

The critical concern of HF is severe soft tissue irritation and the subsequent technical difficulties that arise in clinical situations [22]. Hence, an alternative etchant that is safer and easier to use is 37% phosphoric acid (PA). Moreover, previous studies demonstrated that the zirconia surface tomography and the roughness of PA and HF etchings were not significantly different [23–25].

However, to date, no studies have comprehensively compared the SBS between PA and HF surface treatments in bonding metal brackets to unglazed zirconia in both immediate loading (IML) and after artificial aging by thermocycling (TMC). Therefore, this study aimed to compare the SBS under the conditions of IML and after the TMC of metal brackets to unglazed zirconia treated with either HF or PA etchants. A control group without surface treatment (NT) was also included.

2. Materials and Methods

2.1. Sample Size Calculation

The sample size was determined based on a previous study by Yu et al. [24] to detect the mean SBS differences between groups with 90% power and a significance level of $p < 0.01$ using G*Power version 3.1.9.6. (University of Duesseldorf, Duesseldorf, Germany). The calculation indicated that a minimum of 14 specimens were required for each of the six groups. Since one additional specimen of each surface treatment was needed for scanning electron microscopy (SEM) images, the total sample size was 87 specimens.

2.2. Zirconia Specimen Preparation and Mechanical Pretreatments

Zirconia blanks (BruxZir Shaded 16, Prismatik Dentalcraft, Irvine, CA, USA) were fabricated into 6 mm diameter and 1.5 mm thick cylinders and fully sintered according to the manufacturer's instructions ($n = 87$). They were all embedded in self-cured acrylic resin blocks (UNIFAST™ Trad; GC America, Alsip, IL, USA). The surface of the specimens was finished under water cooling with 240- to 2000-grit sandpaper using a grinder-polisher (MetaServ Lab Grinder-Polisher; Buehler, IL, USA). Fine polishing was performed with a felt polishing disc and a 1-µm diamond suspension (MetaDi® Monocrystalline Diamond Suspension 1 µm 40–6530; Buehler, IL, USA) under a lubricant (MetaDi® Fluid 40–6016; Buehler, IL, USA). All specimens were cleansed with distilled water for one minute prior to further measurements. Surface roughness was measured using a profilometer (Surfcorder SE-2300; Kosaka Laboratory, Tokyo, Japan) to standardize the initial roughness of all specimens. The average value of the arithmetic average roughness (Ra) after three repeated measurements should not exceed the standard of 0.2 µm [26].

Six groups of specimens were defined: the three methods of surface treatment and the TMC of each surface treatment (Figure 1). Stratified sampling was used to ensure that the average Ra of each group was equal (0.007 µm).

- No surface treatment (1. NT, 2. NT + TMC): The specimens were rinsed with distilled water for 30 s and then air-dried.
- HF etchant (3. HF, 4. HF + TMC): The lowest concentration reported in the literature [16,21] of 4% HF (Porc-Etch; Reliance Orthodontic Products, Itasca, IL, USA) was applied to the zirconia surface for 2 min, rinsed with distilled water for 30 s, and then air-dried.
- PA etchant (5. PA, 6. PA + TMC): PA at 37% (3M™ Scotchbond™ Multipurpose Etchant; 3M ESPE, St. Paul, MN, USA) was applied to the zirconia surface for 2 min, rinsed with distilled water for 30 s, and then air-dried.

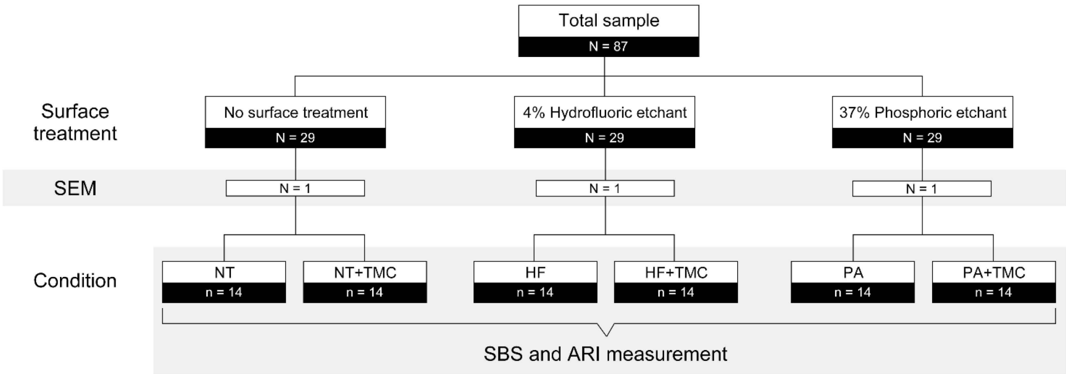

Figure 1. Flow chart of the sample allocation into six groups based on surface treatment methods and treatment conditions. ARI—adhesive remnant index; SBS—shear bond strength; SEM—scanning electron microscopy; TMC—thermocycling.

After acid surface treatment, one specimen from each surface treatment was randomly selected for surface topography examination under SEM (FEI Quanta 400; Thermo Fisher Scientific, Brno-Černovice, Czech Republic) at 500×, 1000×, 2000×, and 4000× magnification.

2.3. Bracket Bonding Procedure

Custom-made waterproof stickers with a puncture mimicking the size of a bracket base were affixed to each of the remaining specimens (n = 14/group) to standardize the bonding area (Figure 2A). The exposed surface was conditioned with 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) containing a bonding agent (Assure® PLUS All Surface Bonding Resin; Reliance Orthodontic Products, Itasca, IL, USA) and was air-dried for 20 s. Mandibular incisor metal brackets (Unitek Gemini Brackets Roth; 3M Unitek Orthodontic Products, Monrovia, CA, USA) were bonded onto the zirconia with a light-cured composite resin (Pad Lock; Reliance Orthodontic Products, IL, USA) (Figure 2B) under a 300 g seating force. After the removal of any excess resin, the specimens were light-cured for 20 s from the occlusal and gingival directions using a light-emitting diode device (Demi™ Plus; Kerr Corporation, Orange, CA, USA). The stickers were then peeled off (Figure 2C). The specimens in the TMC group were thermocycled for 5000 cycles [27] in distilled water at 5 °C and 55 °C for a dipping time of 30 s and a dwell time of 10 s.

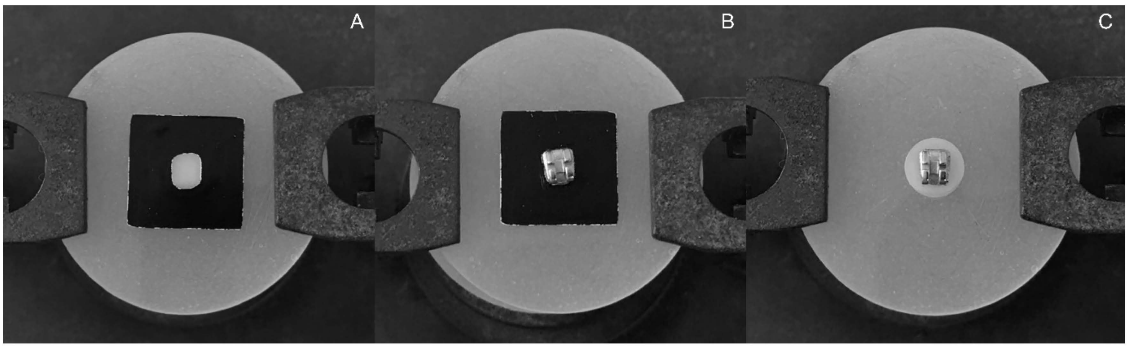

Figure 2. Bracket bonding procedure. (**A**) The bonding area was defined by a custom-made waterproof sticker. (**B**) The bracket was placed onto the zirconia surface. (**C**) After light-curing, the sticker was peeled off.

2.4. Shear Bond Strength Test and Failure Behavior Analysis

The SBS test was performed using a universal testing machine (Lloyd Instruments LRX-Plus; AMETEK Lloyd instruments, Hampshire, UK) (Figure 3) with a 250-N loading cell at a crosshead speed of 0.5 mm/min until failure. The failure mode was photographed at 40× magnification under a 3D profilometer (Keyence VR6200; Keyence Corporation, Osaka, Japan) and classified based on the adhesive remnant index (ARI) by Bishara et al. [28] using ImageJ software (Figure 4A–E).

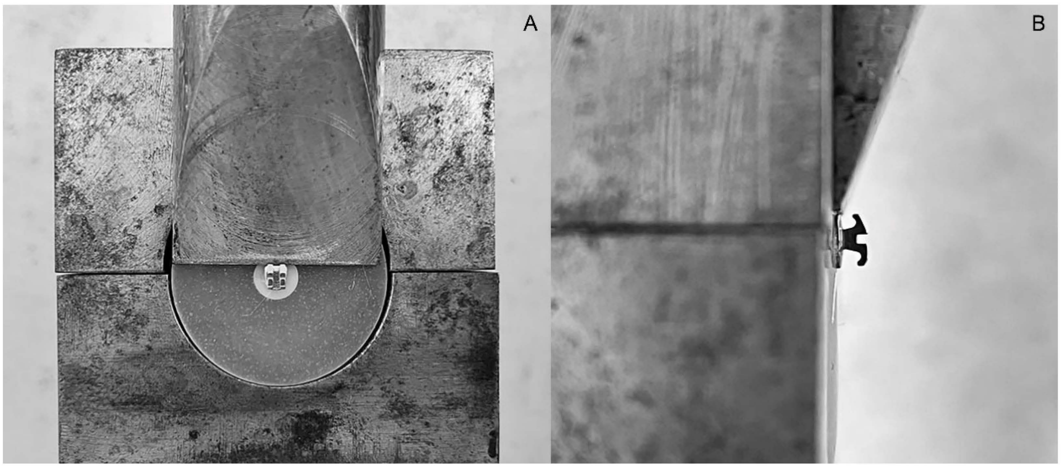

Figure 3. (**A**,**B**) Shear bond strength test with the universal testing machine.

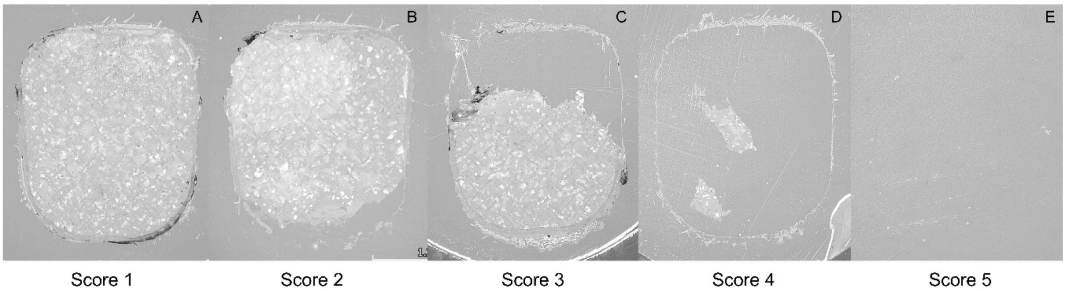

Figure 4. Adhesive remnant index (ARI) score 1–5 at 40× magnification under a 3D profilometer. (**A**) Score 1: All of the resin remained on the zirconia and showed a bracket base impression. (**B**) Score 2: Equal to or more than 90% of the resin remained on the zirconia. (**C**) Score 3: Between 10% and 90% of the resin remained on the zirconia. (**D**) Score 4: Less than 10% of the resin remained on the zirconia. (**E**) Score 5: No resin remained on the zirconia surface. This figure used samples from this study to illustrate the amounts of adhesive remnant after bracket removal. The scoring followed the index reported in a study by Bishara et al. [28].

2.5. Statistical Analyses

The Kolmogorov–Smirnov test showed that the SBS data were normally distributed; however, the ARI data were not normally distributed. The differences of SBS within and between surface treatment groups were assessed with an independent *t*-test and one-way ANOVA, followed by the Scheffe post hoc test. The differences in the ARI scores within and between groups were evaluated by the Mann–Whitney and Kruskal–Wallis tests, respectively. All statistical analyses were set at $p < 0.01$.

3. Results

The SEM images of the zirconia surfaces after the 2 min acid etching are shown in Figure 5. The untreated zirconia (NT) exhibited a smooth surface, whereas the HF and PA etched groups showed islands of tiny porosities in all magnifications. HF demonstrated more prominent irregularities than the PA.

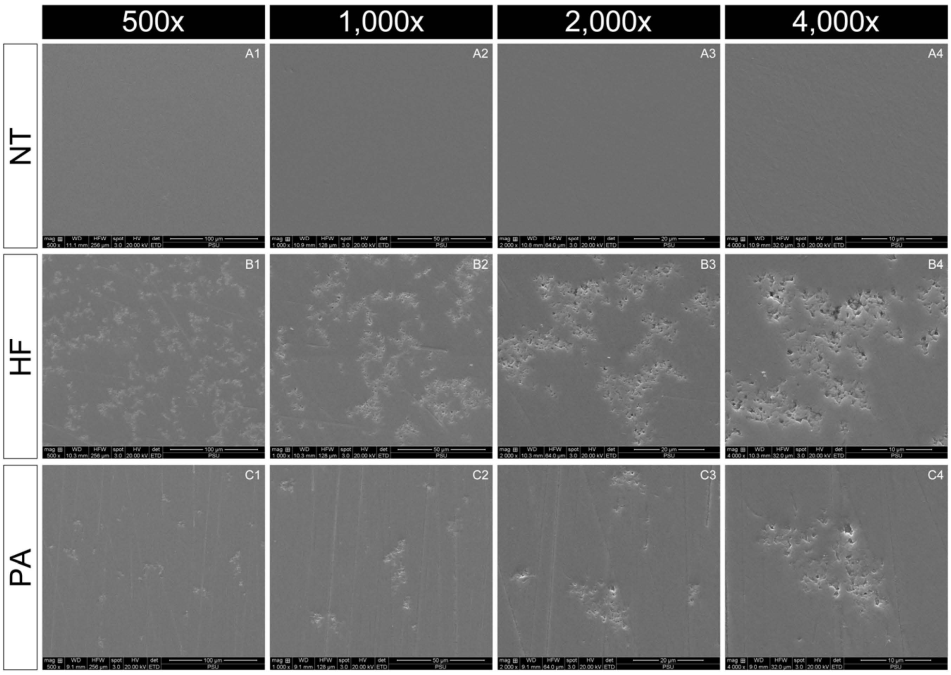

Figure 5. Zirconia surface topography under scanning electron microscopy at (**1**) 500×; (**2**) 1000×; (**3**) 2000×; and (**4**) 4000× magnification. (**A**) Specimen had no surface treatment (NT). (**B**) Specimen was etched with 4% hydrofluoric etchant (HF). (**C**) Specimen was etched with 37% phosphoric etchant (PA).

Table 1 shows that TMC resulted in a significantly lower mean SBS than IML in all of the acid surface treatment groups (NT, HF, and PA; $p < 0.01$). Considering the TMC/IML percentages, the ratios of NT + TMC/NT, HF + TMC/HF, and PA + TMC/PA were 53.45%, 42.63%, and 42.74%, respectively. NT showed the highest mean SBS in both the IML and TMC conditions, followed by PA and HF. However, the differences between the different surface treatments under the same conditions were not statistically significant ($p > 0.01$).

Table 1. Comparisons of the shear bond strength between different the acid surface treatments in immediate loading and thermocycling.

Etchants	Shear Bond Strength (MPa) Immediate Loading	Shear Bond Strength (MPa) Thermocycling	*p*-Value * (Within Groups)	Mean Differences (MPa)	% Difference TMC/IML (%)
NT	8.98 ± 3.66 [a]	4.80 ± 1.62 [a]	0.001	4.18 ± 1.07	4.80/8.98 (53.45)
HF	7.67 ± 2.68 [b]	3.27 ± 2.17 [b]	<0.001	4.40 ± 0.92	3.27/7.67 (42.63)
PA	8.40 ± 2.27 [c]	3.59 ± 1.75 [c]	<0.001	4.81 ± 0.77	3.59/8.40 (42.74)
p-Value ** (Between groups)	0.50	0.85			

* Difference within groups by independent *t*-test at $p < 0.01$. ** Difference between groups by one-way ANOVA test at $p < 0.01$. HF—4% hydrofluoric etchant ($n = 14$); IML—immediate loading; NT— no surface treatment ($n = 14$); PA—37% phosphoric etchant ($n = 14$); TMC—thermocycling. The same characters (a, b, c) indicate the significant differences found when comparing the etchants in either immediate loading or thermocycling.

After bracket debonding (Table 2), the median ARI score after TMC was significantly higher than the IML in all acid surface treatment groups ($p < 0.001$). However, there were no significant differences in the median ARI scores between the different surface treatments in either the IML or TMC condition ($p = 0.01$).

Table 2. Comparisons of the adhesive remnant index (ARI) scores between different acid surface treatments in immediate loading and thermocycling.

	ARI Score, Median (IQR)		p-Value * (Within Groups)
Etchants	**Immediate Loading**	**Thermocycling**	
NT	2.00 (1.00) [a]	3.00 (1.00) [a]	<0.0001
HF	3.00 (1.25) [b]	4.00 (0.25) [b]	<0.0001
PA	3.00 (1.00) [c]	4.00 (0.00) [c]	<0.0001
p-Value ** (Between groups)	0.01	0.01	

* Differences within groups by Mann–Whitney test at $p < 0.01$. ** Difference between groups by Kruskal–Wallis test at $p < 0.01$. HF—4% hydrofluoric etchant (n = 14); IQR—interquartile range; NT—no surface treatment (n = 14); PA—37% phosphoric etchant (n = 14). The same characters (a, b, c) indicate significant differences found when comparing the etchants in either immediate loading or thermocycling.

4. Discussion

This study demonstrated that the mean SBS results were acceptable for orthodontic bonding only in the IML condition (7.67 MPa to 8.98 MPa) in all surface treatment groups. Unfortunately, after TMC, all surface treatment groups showed weak mean SBS values (3.27 MPa to 4.80 MPa) that were less than the acceptable range [7,8]. Therefore, the results implied that etched zirconia under the in vitro artificial aging protocol provided insufficient bond strength for orthodontic bonding.

Mehmeti et al. [19] compared the bond durability between the HF and PA etched on glazed zirconia. Although the glazing technique, which contained silica [29], enhanced the resin–zirconia bond after acid etching, a higher wear of opposing enamel was observed [30,31]. Therefore, this study was conducted using unglazed zirconia due to its wear-friendliness and comparable aesthetics in clinical scenarios [32]. The crucial confounder that could interfere with the results of the experiment was the initial surface roughness of zirconia [33]. This study controlled the initial surface roughness of each zirconia prior to the SBS test.

This study confirmed that the zirconia surface, despite being a silica-free material [3,6] was altered by acid treatment. Callister and Rethwisch [34] explained that atoms located at the grain peripheries, which exhibit a higher chemical reactivity than atoms in the center, dissolved more rapidly. This process possibly enlarged the inter-grain gaps that led to the formation of porosities as the grains dislodged [18].

Untreated zirconia presented the strongest mean SBS, followed by PA and HF without significant differences. These findings were supported by previous studies [35,36]. The explanation is that acid-induced pores were not necessarily an optimal retentive archetype for mechanical interlocking [21,37]. High-viscosity resin encountered difficulty penetrating such deep and small pores [18,38], which resulted in a reduced effective bonding surface area.

Thermocycling is a widely adopted artificial aging method for bond durability testing on resin-based materials [11,39,40]. This method challenges the bond strength through hydrolysis and thermal stress that represents moisture and temperature fluctuations during meals [12,39]. Gale and Darvell [27] proposed that 10,000 cycles approximated a year of material exposure in the oral environment. Therefore, the 5000 cycles used in this study represented the first six months after bracket placement, which is generally associated with the initial phase of orthodontic treatment.

The mean SBS was significantly lower in the TMC conditions in all surface treatments compared to the IML, which was consistent with previous studies [11,12,21]. The untreated zirconia showed a 53.45% decrease in the mean SBS, which corresponded with a study by Inokoshi et al. [11] that reported around 50% or less reduction after TMC when zirconia received no mechanical surface treatment. Moreover, the HF + TMC/HF and PA + TMC/PA ratios showed more reduction in the mean SBS than the NT + TMC/NT. The greater drop in both acid-treated groups was probably due to the smaller bonding surface areas between the resin and zirconia, as mentioned above. Consequently, greater interface degradation occurred during TMC [39].

The results revealed that all surface treatments after TMC failed to provide an efficient bond strength for bracket bonding. This study revealed very weak mean SBS compared to Menezes et al. [36]. They reported 17.99 ± 2.92 MPa in the untreated group and 16.87 ± 1.69 MPa in the HF-etched group between the cylindrical resin cements and zirconia. However, the micro-SBS test used in their study possibly contributed to the higher bond strength than reported in our study [41].

The essential factors for enhancing bond durability between zirconia and resin include creating a micro-roughened zirconia surface, expanding the bonding area, adjusting the surface energy, and improving wettability [42]. This study polished the zirconia to achieve a surface roughness (Ra) of 0.007 µm per group that followed the recommendation of Bollen et al. [26]. Maintaining a surface roughness below 0.2 µm is crucial in preventing plaque accumulation [26], and well-polished zirconia reduces the risk of tooth abrasion that otherwise occurs with a rough zirconia surface. Unfortunately, the polished zirconia in this study failed to generate sufficient roughness and irregularities to establish effective interlocking between the resin and zirconia ceramic, which consequently diminished the shear bond strength between the resin and zirconia.

The introduction of a zirconia surface treatment for optimal bonding was introduced by Kern et al. [43]. A resilient resin bond to zirconia was attained solely through sandblasting the ceramic and utilizing a resin composite containing a specific phosphate monomer. The MDP facilitates a water-resistant chemical bond to zirconia with the phosphate ester group of the monomer binding directly to the metal oxides of zirconia. Therefore, the surface roughening and activation via air abrasion with aluminum oxide particles before adhesive bonding, along with the utilization of a resin composite containing MDP, are imperative to achieving a durable bond to densely sintered zirconia ceramics [44–47].

The assessment of bond quality in this study was not only based on bond strength but also considered the failure mode. Della et al. [48] recommended this protocol because it reveals crucial information on the clinical performance limits in bonding systems. Irrespective of the types of surface treatment, the ARI analysis demonstrated that most failures were the mixed failure type that indicated both adhesive and cohesive failure. This result was similar to previous studies [19,25]. After TMC, the median ARI significantly shifted towards the higher scores, which suggested a weakened resin–zirconia bond through the artificial aging procedure. This confirmed that the adhesive bond between the resin–zirconia interface was indeed degraded by TMC [12,21,38].

Some limitations need to be taken into account. The laboratory results might not completely reflect the clinical setting since the oral environment is complex and cannot be recreated in vitro [49]. Moreover, the use of zirconia blanks and bracket bonding in our study was limited to unglazed zirconia that was finely polished and may not perfectly represent clinical practice. The dynamic occlusal force, which differs from a static shear force induced by the testing machine, also affects the bond strength. This experiment was performed on a flat zirconia surface; however, the intraoral zirconia surface is generally composed of curvatures and grooves to mimic the contour of a natural tooth. This study was limited to only two types of acid surface etching. Therefore, comprehensive testing to include a comparison with standard treatment, such as sandblasting, should be further examined. Moreover, other bracket materials and other surface treatments are recommended for further investigation. Finally, clinical trials are necessary to prove these laboratory findings.

5. Conclusions

In conclusion, TMC presented SBS values that were 42.63–53.45% lower than IML. Two-minute simple etching methods with either 4% HF or 37% PA produced insufficient SBS of metal brackets to zirconia in orthodontic practice after TMC. The ARI score showed mixed failure after debonding between the metal brackets and zirconia.

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Data Availability Statement: Data are available upon request.

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