ORIGINAL RESEARCH

Evaluation of Different Surface Roughening Techniques on Clear Aligner Attachments Bonded to Monolithic Zirconia: *In Vitro* Study

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Abstract

Aim: The objective of this study is to assess the impact of three different surface etching techniques and two composite materials on surface roughness (R_a) and the shear bond strength (SBS) of clear aligner attachments bonded to monolithic zirconia (MZ).

Materials and methods: Sixty-six MZ disks were divided into three main groups (n = 22) according to the surface treatment method: group I: hydrofluoric acid (HFA) 9.5%, group II: 50 μm aluminum oxide (Al_2O_3) sandblasting, and group III: Er:Cr:YSGG laser. Each group was then subdivided into two subgroups according to the composite material (n = 11) used for bonding of the attachment, either packable composite Filtek Z350 or composite Filtek Z250. Clear aligner attachments were then bonded to the disks, and measurements of R_a and SBS were taken. Statistical analysis was carried out using the Kruskal–Wallis test and two-way analysis of variance.

Results: The HFA groups showed the highest SBS (11.29 ± 2.83 MPa) and R_a (0.82 μm), while the laser groups exhibited the lowest SBS (8.29 ± 2.38 MPa) and R_a (0.634 μm). In Filtek Z350, there was a significant difference observed between the subgroups regarding SBS but not in Filtek Z250 subgroups.

Conclusion: The 9.5% HFA provided the highest SBS value in comparison with Er:Cr:YSGG laser and sandblasting (p <0.05).

Clinical significance: This study has the potential to benefit both orthodontists and patients by providing insights into techniques that can improve the bond strength and longevity of clear aligner attachments to MZ.

Keywords: Clear aligner, Composite attachment, Monolithic zirconia, Shear bond strength, Surface treatment.

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Introduction

Due to the increasing esthetic demands of individuals, there has been a corresponding rise in the use of removable clear aligners. Align Technology launched Invisalign in 1998, which consists of a series of removable thermoplastic aligners. Clear aligner therapy offers distinct advantages, such as improved esthetics, the ability to be removed, and reduced dimensions, compared with conventional fixed appliances.[1] However, relying solely on aligners without auxiliary aids is associated with several limitations. Clear aligner approach is restricted to pushing teeth into pre-existing spaces, directing them far from the center of resistance.[2]

The demand of patients has motivated manufacturers to pursue innovative strategies to enhance the effectiveness of their products. This includes the incorporation of auxiliaries, such as composite attachments, bite ramps, precise cuts, and power ridges. These enhancements enable the management of a wide range of malocclusions using aligners.[3] Through the application of force closer to the center of resistance, this results in a more control and uniform tooth movement.[4]

Consequently, composite attachments have become an essential element of aligner treatment. Even with their integration, successfully accomplishing the intended teeth movement as planned remains a clinical challenge.[5]

Several techniques have been employed to enhance the bonding of resin materials to zirconium ceramic, including acid etching, airborne particle abrasion, and tribochemical silica application.[6,7] Air particle abrasion enhances the adhesion capacity and improves the surface tension and wettability of a prepared microretentive zirconium surface, thus promoting the formation of a strong cement–ceramic micromechanical link.[8]

Kunt and Duran and others support utilizing laser irradiation as a substitute technique for surface treatment of restorations made of ceramic, resin matrix, or composite materials.[9–11] This approach is designed to achieve micromechanical adhesion by augmenting the surface roughness of the restoration. For this purpose, many researchers choose laser types like erbium-doped yttrium aluminum garnet (Er:YAG), neodymium garnet yttrium aluminum (Nd:YAG), and erbium, chromium: yttrium-scandium-gallium-garnet (Er:Cr:YSGG).

There is lack of evidence-based protocols and limited research in clear aligner attachment bonding to monolithic zirconia (MZ).

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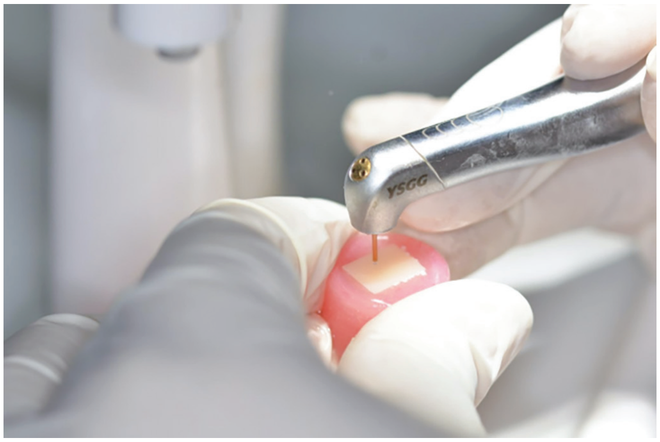

Fig. 1: Zirconia roughening by Er:Cr:YSGG laser

Therefore, the aim of this study was to assess different surface conditioning techniques and composite materials on shear bond strength (SBS) of clear aligner attachments bonded to MZ.

Materials and Methods

This *in vitro* study was approved by the Ethical Committee of Mansoura University, Mansoura, Egypt (code no: A02010240R). This study was done in 2024 for 6 months (March to September).

Sample Size

Using Power Analysis and Sample Size software (version 2017) (Kaysville, Utah, USA), it was found that the preparation of 66 samples was sufficient to obtain a power of 82% and a significance level of 0.05 effect size $f(\sigma m/\sigma) = 0.4000$.

Creation of Groups

Sixty-six MZ ($10 \times 5 \times 3$ mm^3) (Zolid HT+ white) (Amann Girrbach AG, Koblach, Austria) blocks were secured by being fixed into equal-sized acrylic blocks. They were divided equally (n = 22 specimens) into three main groups based on the surface treatment methods employed. Subsequently, each group was divided into two subgroups (n = 11) based on composite attachment material applied.

Surface Treatment Groups

Group I: Roughening was done using 9.5% hydrofluoric acid (HFA) for 2 minutes in a gel base form (Bisco, USA).

Group II: Sample surfaces were sandblasted with 50-µm Al_2O_3 utilizing an air-abrasion laboratory device (Renfert GmbH, Germany). The procedure was subjected to a pressure of 40 psi for a duration of 5 seconds at a 5 mm distance.

Group III: Roughening was done by Er:Cr:YSGG laser (Waterlase, CA, USA) of 2,780 nm wavelength with the power of 4 W, mode of frequency 15 Hz, and ratio of 55% water and 65% air (Fig. 1).

Following surface treatment, all disks were thoroughly cleaned with tap water for more than 30 seconds, and they were then allowed to air dry for 30 seconds.

Surface Roughness Test

Before bonding, the mean surface roughness value (R_a) of all groups was assessed using a profilometer (Surftest SJ 201, Kawasaki, Japan). The stylus passed each specimen in a reciprocating motion, obtaining three readings, with a scanning speed of 0.5 mm/second.

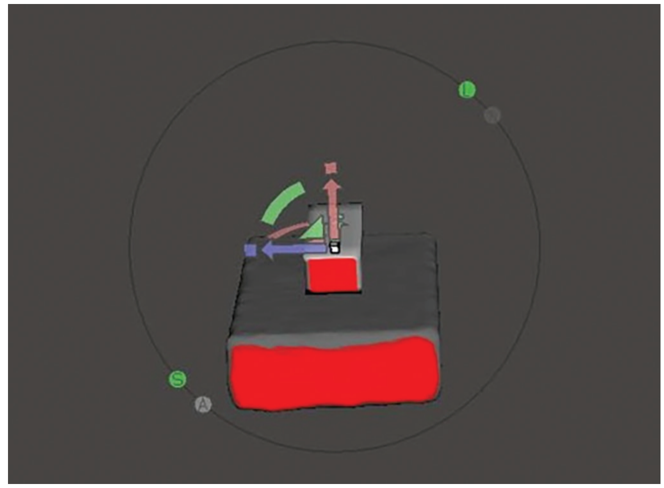

Fig. 2: Attachment design using Exocad software

Attachment Bonding Stage

The following dimensions were used to create a rectangular composite attachment template: a height of 4 mm, width of 2 mm, and prominence of 1 mm designed by Exocad software (Fig. 2). A 0.6-mm thick, vacuum-formed aligner sheet (Scheu-Dental Q5 GmbH, Germany) was used to create the composite attachment template using Biostar® pressure-molding equipment (Iserlohn, Germany).[12]

Z-PRIME (Bisco Products, USA) was applied after the roughening processes.

The bonding process involved the use of a Single Bond Universal Adhesive (3M, ESPE, USA). Two different composites (Fitlek Z250 and Fitlek Z350 3M ESPE, Monrovia, CA, USA) were used to fill the attachment space in the template and then positioned onto the MZ disks. The attachments were cured for 3 seconds using Woodpecker LED device (Guilin Woodpecker Co., China). After curing, the template was removed.

Thermocycling Stage

All samples underwent thermocycling for 1,000 cycles (ranging from 5 to 55°C) with a 15-second waiting interval and a 10-second waiting time using T100 SD mechatronic thermocycler (Germany).

SBS Test

The SBS data was measured at a crosshead speed of 1 mm/minute using the Universal Testing Machine (Model 3345, England). The readings were repeated for the second and third time at 2-weeks interval. After being recorded in newtons (N), the data were converted to megapascals (MPa).

Statistical Analysis

Data analysis was performed by Statistical Package for the Social Sciences software, version 25 (PASW Statistics, SPSS Inc., Chicago). Following the Kolmogorov–Smirnov test to determine normality, quantitative data had been represented using the median for nonnormally distributed data (R_a) and the mean ± standard deviation for normally distributed data (SBS). The significance of the results was evaluated at the ≤0.05 level. To assess the differences in R_a between two or more groups under investigation, the Mann–Whitney U-test and Kruskal–Wallis test were independently performed. A Student's t-test was employed

Table 1: Reliability of the surface roughness and SBS between readers

	Surface roughness			*SBS*		
		95% confidence interval			*95% confidence interval*	
	Intraclass correlation	*Lower bound*	*Upper bound*	*Intraclass correlation*	*Lower bound*	*Upper bound*
Readings 1 and 2	0.967	0.946	0.980	0.977	0.962	0.986
Readings 1 and 3	0.957	0.930	0.974	0.996	0.994	0.998
Readings 2 and 3	0.993	0.988	0.996	0.972	0.955	0.983

Table 2: Surface roughness (R_a) among studied groups

Surface roughness	*HFA*	*Sandblast*	*Laser*	*Test of significance*
Median	0.82	0.673	0.634	KW = 25.86
Minimum	0.29	0.39	0.13	*p* = 0.001*
Maximum	1.6^{A}	$1.59A^{B}$	0.99^{B}	

KW, Kruskal–Wallis test; *Statistically significant. Similar superscripted letters denote nonsignificant difference between groups within same row

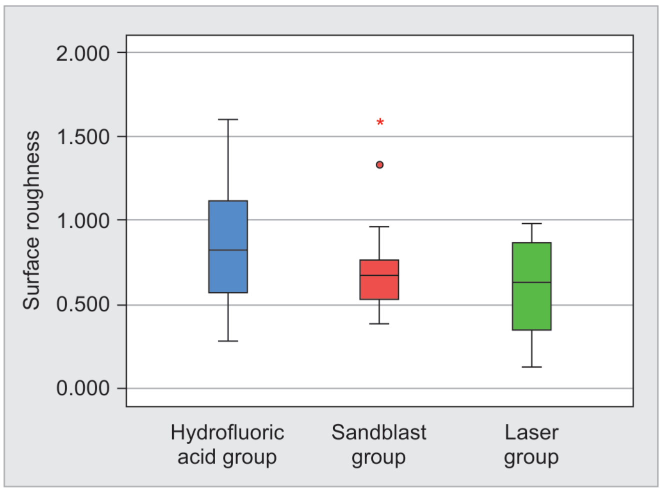

Fig. 3: Box and Whisker plot showing R_a among studied groups

to compare two independent groups for SBS. Several independent groups were evaluated using a one-way analysis of variance (ANOVA) test, and pairwise comparisons were made using a *post-hoc* Tukey test. A two-way ANOVA test was employed to evaluate several combinations of two independent factors on a continuous outcome.

Results

Table 1 illustrates excellent agreement between readers in surface roughness and SBS. Excellent agreement (intraclass correlation coefficient = 0.946–0.996) was found between the three readings.

The median R_a value was highest among the HFA group, followed by the sandblast group, and then the laser group (0.82, 0.673, and 0.634, respectively) (Table 2 and Fig. 3). Pairwise comparison shows a statistically significant difference between the HFA and laser group ($p = 0.016$).

For Filtek Z250 composite, Table 3 revealed no statistically significant difference between the studied groups in term of SBS ($p = 0.097$). The mean SBS was highest among the HFA group, followed by the sandblast group, and then the laser group (10.57 ± 2.49, 10.31 ± 2.89, and 8.29 ± 2.38, respectively). *Post-hoc* Tukey test was used for pairwise comparison and shows a statistically significant difference between the HFA and laser groups ($p = 0.049$). No statistically significant difference was found between the HFA and sandblast groups ($p = 0.811$) and between the laser and sandblast groups ($p = 0.08$).

The Filtek Z350 composite showed a statistically significant difference between the studied groups in terms of SBS ($p = 0.019$). The mean SBS was highest among the HFA group, followed by the sandblast group, and then the laser group (11.29 ± 2.83, 10.59 ± 2.76, and 8.33 ± 1.37, respectively). Pairwise comparisons revealed statistically significant differences between the HFA and laser groups ($p = 0.007$) and between the laser and sandblast groups ($p = 0.036$). No statistically significant difference was found between the HFA and sandblast groups ($p = 0.509$). There was also no statistically significant difference found between composites Z250 and Z350 within the HFA ($p = 0.539$), sandblast ($p = 0.814$), and laser ($p = 0.971$) groups.

The interaction between the HFA and Filtek Z350 exhibited the highest SBS (11.29 ± 2.83 MPa), followed by the interaction between sandblast and Filtek Z350, while the interaction between the laser and Filtek Z250 demonstrated the lowest (8.29 ± 2.38 MPa).

Discussion

Clear aligner therapy has recently gained popularity among adults with ceramic restorations. As a result, the most acceptable approach for ceramic surface treatment in clinical practice has been debated. Although multiple techniques have been given for bracket bonding to MZ ceramics, there is minimal data on aligner attachment bonding to various ceramics.[13–16]

Choosing the appropriate material for clear aligner composite attachment remains a major challenge for orthodontists. Several studies have examined the effect of low- and high-viscosity composites on SBS of clear aligner attachment.[17,18] They found that high-viscosity composites had higher SBS than low-viscosity composites. Moreover, they claimed that the amount of inorganic filler in the composite affects SBS. Consequently, in this study, two different high-viscosity composites were chosen. Nevertheless, a statistically significant difference could not be found, which goes consistent with Silva et al.[19] Furthermore, Santos et al. stated that there is no significant difference between Z250 and Z350 in terms of SBS.[20]

Thermal cycling creates a direct link between the expansion of the restorative material and the tooth, mimicking the temperature changes in the oral cavity. The effect of thermal cycling is influenced by three key factors: Temperature, holding duration, and number of cycles.[21] The ISO-TR 11450 standard recommends conducting a thermal cycling test for artificial aging, involving subjecting the material to 500–10,000 cycles in water baths with temperatures ranging from 5 to 55°C.[21,22] The specimens utilized in the investigations were subjected to multiple temperatures for different durations, including 10, 15, 30, 55, and 60 seconds, as well as 2 and 3 minutes.[23] In this investigation, the specimens underwent

Table 3: Comparison of SBS of two different clear aligner attachment materials (composite Z250 and Z350) bonded to MZ using three different roughening techniques

Shear bond strength	*HFA* *Mean ± SD*	*Sandblast* *Mean ± SD*	*Laser* *Mean ± SD*	*F*	*p-value*
Composite Z250	10.57 ± 2.49[a]	10.31 ± 2.89	8.29 ± 2.38[a]	2.52	0.097
Composite Z350	11.29 ± 2.83[a]	10.59 ± 2.76[ab]	8.33 ± 1.37[b]	4.51	0.019*
Test of significance	$t = 0.625$	$t = 0.239$	$t = 0.037$		
	$p = 0.539$	$p = 0.814$	$p = 0.971$		

F, one-way ANOVA test; *Statistically significant. Similar superscripted letters in same row denote significant difference between groups by *post-hoc* Tukey test

1,000 cycles of thermal cycling, ranging from 5 to 55°C. Each cycle consisted of a 15-second waiting time and a 10-second transfer period. Consistent with this research, numerous other studies have utilized 1,000 cycles of thermal cycling.[23–26]

Zirconia is the strongest and toughest of all ceramics. To bond composite attachment to zirconia with adequate SBS, zirconia must be treated using a variety of procedures, including mechanical and chemical treatments using strong acids. As a result, several investigations have shown that the SBS test is an appropriate approach for determining the adhesion strength of composite resins to enamel.[27,28]

Previous studies have proven that employing HFA may not provide sufficient bond strength.[29,30] However, in this study, HFA etching produced the highest R_a mean value in contrast to other surface treatments. Our result goes consistent with Liang et al. who found that HFA had the highest R_a value in comparison with sandblasting and HCl groups.[31] In this study, the least R_a value was found in Er:Cr:YSGG group, which is in total agreement with Dikicier et al.[32] Furthermore, subgroups (Z350 and Z250) treated with HFA exhibited higher SBS than those treated with sandblasting and laser. These results are in harmony with Zhang et al.[33]

A variety of studies have examined the effects of HFA with varying doses as an etchant on zirconium.[23,34–37] The justification for using HFA acid to etch the surface is that the zirconia is not in its original state since it has been altered by the addition of other oxides (ZrO_2 + HfO_2 + Y_2O_3: ≥99.0, Y_2O_3: 6.7–7.2, HfO_2: ≤5, Al_2O_3: ≤0.5, and other oxides: ≤1). The manufacturing company may be related to the enhanced translucency and strength of this type in contrast to the other types. The effect of HFA conditioning on the micromorphology of various types of glassy ceramics differs and depends not solely on their chemical composition but on their microstructural pattern also.[38] Recent studies reported that it can induce changes on the zirconia surface.[39,40]

Various researches have shown the beneficial impact of abrasion using airborne particles on the surface of restorations, as aluminum oxide particles (Al_2O_3) could potentially affect their surface.[23,41] In our study, the sandblast-treated group showed the clinically accepted SBS, although it was lower than the group treated with HFA. This difference can be explained by the different grain sizes we used; this aligns with El-Wahab et al.[42]

The results of this study demonstrated that the highest mean SBS is achieved by the interaction of HFA and Z350, while the lowest SBS was achieved by the interaction of laser and Z250. In the current study, all groups showed SBS values that are consistent with Hobson et al.[43]

The results of the current study show that using the Er:Cr:YSGG laser for surface treatment of the zirconia created a significantly enhanced SBS similar to Zeidan et al.[44] This enhancement can be explained by the fact that laser applications remove particles through microexplosion, a process known as ablation. The Er:Cr:YSGG laser-induced roughness increases the potential bonding area of zirconia.[45] Saade et al. evaluated various surface conditionings on the bonding between resin and zirconia and found that the Er:Cr:YSGG laser was an effective approach for noninvasive surface roughening technique.[46] However, in comparison with HFA and sandblasting, it exhibited the least findings, attributable to several aspects, including laser parameters such as wavelength, power, density, and pulse duration. This aligns with Ghoveizi et al.[47] Comparison of laser and sandblasting demonstrated a lower SBS value of laser. Nonetheless, further clinical and *in vitro* investigations are needed on the surface parameters of dental ceramics, as there remains insufficient data on the optimal laser type and application methods.

However, this research has some limitations that need acknowledgment. The experiments were conducted *in vitro*, which may not entirely reflect the complexities of clinical scenarios. Consequently, future research must validate the *in vitro* trial results by comprehensive clinical studies. The SBS of composite attachments when bonded to different zirconia materials must be further investigated.

Conclusion

Based on limitations of the *in vitro* study, we have concluded that roughening with 9.5% HFA resulted in the highest R_a and SBS in both types of composites. The Filtek Z350 composite demonstrated higher SBS compared with the other group. The laser roughening technique showed the lowest R_a and SBS values, indicating a need for further investigation with varying parameters.

Ethical Approval

The present study was approved by the Ethical Committee of Mansour University (code no: A02010240R).

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