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ORIGINAL ARTICLE

Bond strengths of brackets bonded to enamel surfaces conditioned with femtosecond and Er:YAG laser systems

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Abstract The aim of this study was to compare femtosecond and Er:YAG laser systems with regard to enamel demineralization and bracket bond strength. Human-extracted premolars were randomized to three groups ($n = 17$) depending on the conditioning treatment used for the buccal surfaces: 37 % orthophosphoric acid, Er:YAG laser etching (MSP mode 120 mJ, 10 Hz, 1.2 W), and femtosecond laser etching (0.4 W, 800 nm, 90 fs/pulse, 1 kHz). Metal brackets were bonded with Transbond XT to the conditioned surfaces and light cured for 20 s. The samples were thermocycled (5000 cycles, 5–55 °C) and subjected to shear bond strength (SBS) testing using a universal testing machine. Failure types were analyzed under an optical stereomicroscope and SEM. The adhesive remnant index (ARI) was evaluated to assess residual adhesive on the enamel surface. The results revealed no significant differences in SBS between the Er:YAG laser (7.2 ± 3.3 MPa) and acid etching groups (7.3 ± 2.7 MPa; $p < 0.05$), whereas a significant difference was observed between the femtosecond laser etching group (3.3 ± 1.2 MPa) and the other two groups ($p < 0.01$). ARI scores were significantly different among the three groups. The results of our study suggest that laser conditioning with an Er:YAG system results in successful etching, similar to that obtained with acid. The sole use of a femtosecond laser system may not provide an adequate bond strength at the bracket–enamel interface.

Keywords Femtosecond laser · Er:Yag laser · Enamel surface conditioning · Shear bond strength · Enamel demineralization

Introduction

Ideal bonding in orthodontic practice strongly depends on the bonding procedures used. Therefore, improvements in bonding procedures can lead to decreased bond failure rates, minimal enamel damage, and decreased chairside time. The bond strength at the bracket–enamel interface is influenced by the adhesive material or bonding resin, bracket mesh base design, and conditioning treatment used for the enamel surface [1, 2].

Enamel conditioning methods have important effects on the adhesion of bonding resins. These include acid etching, laser treatment, and sandblasting [3].

The concept of enamel surface etching with phosphoric acid was first proposed by Buonocore in 1955 to increase the bond strength between composite resin and etched enamel [4]. The application of 37 % phosphoric acid is the most common technique for enamel conditioning [5, 6]. However, although acid etching provides high bond strength values, this technique is associated with several disadvantages. Although gel acids are more stable than liquid acids, there is always a shift in the acid on the enamel surface that increases the caries susceptibility of the enamel and increases enamel demineralization [7, 8]. Moreover, the irritating taste during the bonding procedure causes patient discomfort. Laser etching can overcome these disadvantages and has become one of the most

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promising alternatives for enamel surface conditioning. Furthermore, the effectiveness of the adhesive used for bracket bonding has been reported to be equivalent with acid etching and laser etching [9, 10]. With advances in technology, different types of laser systems, including CO_2, Nd:YAG, Er:YAG, and femtosecond laser systems, have been developed and used for enamel conditioning in orthodontic practice [9, 11–13].

In recent years, the use of femtosecond laser systems for enamel surface conditioning has seen a gradual increase. Kabas et al. [14] reported that femtosecond lasers can provide an adequate bond strength with the removal of less dental tissue compared with conventional acid etching techniques. Despite the advantages, femtosecond laser system high cost remains a disadvantage [15].

The adequacy of laser etching with respect to bond strength values remains controversial [9, 12, 13]. To our knowledge, only one study has compared femtosecond laser and Er:YAG laser systems with regard to shear bond strength (SBS) values for metal brackets bonded to enamel surfaces [9].

The aim of this in vitro study was to evaluate the differences between Er:YAG and femtosecond laser systems with regard to enamel demineralization and SBS values for brackets bonded to enamel surfaces. The null hypothesis was that there are no differences in SBS values for brackets bonded to enamel surfaces conditioned with Er:YAG and femtosecond laser systems.

Materials and methods

A power analysis was performed (G*Power software ver. 3.1.10, Franz Faul, Üniversität Kiel, Germany, http://www.psycho.uniduesseldorf.de/aap/projects/gpower, 15.12.2009) to calculate the sample size required for three groups (acid etching, Er:YAG laser system, femtosecond laser system). The results indicated an actual power value of 80 for an effect size of $f = 1$, $\alpha = 005$, power of 94, noncentrality parameter of 15, and critical t value of 5. A requirement of 17 specimens in each group was determined. Accordingly, 51 human extracted maxillary premolars were used in the present study.

Sample preparation

All premolar teeth used in the present study were extracted for orthodontic treatment purposes. Teeth with intact enamel were selected. The exclusion criteria were as follows: pretreatment with chemical agents and the presence of cracks, restorations, infections, and/or enamel defects. All eligible premolars were cleaned with a periodontal scaler for the removal of organic debris on and around the teeth. Then, the teeth were stored in distilled water at room temperature until the beginning of the experiments.

For the experiments, the teeth were embedded in self-cured acrylic blocks, with the buccal surfaces oriented parallel to the force direction used during SBS testing. Each buccal surface was polished for 15 s with nonfluoridated pumice and rubber cups at a low speed (3000 rpm), washed for 30 s, and dried with a water spray for 10 s.

Etching and bonding procedures

Specimens were randomly divided into three groups ($n = 17$) depending on the etching procedure used.

Group 1 was the acid etching group (C). The enamel surfaces were etched with 37 % orthophosphoric acid gel (3M™ ESPE™ Scotchbond™, 3M ESPE, St. Paul, MN, USA) for 20 s, rinsed for 10 s, and dried for 10 s until they appeared frosty white.

Group 2 was the femtosecond laser etching group. Here, a femtosecond laser system was used for surface conditioning. The laser system comprised a Ti:sapphire oscillator (Ti:Light) pumping an ultrafast amplifier (Integra-C), both from Quantronix Corporation (Quantronix Integra-C-3.5, NY, USA). The Ti:Light oscillator produces laser pulses at an 800-nm wavelength with a 90-fs duration at a 80-MHz pulse repetition rate, and pulses from the Integra-C are delivered at a 1-kHz pulse repetition rate at 800 nm with a 90-fs pulse duration and average power of 0.4 W. The laser power is controlled using a neutral density circular filter, and the surface is machined using a Q-Mark system (Quantronic, NY, USA). An 11.5-cm focal length f-θ lens was used to focus the laser beam on a focal spot size measured to be 28 μm in diameter. The laser pulse energy was 400 μJ, and corresponding laser pulse energy density (fluence) was 64.7 J/cm^2 which gives us a 7.19×10^{14}-W/cm^2 laser intensity at the focal point.

Group 3 was the Er:YAG laser etching group. Here, the enamel surfaces were etched with an Er:YAG dental laser (2940 nm wavelength; LightWalker, Fotona, Slovenia) corresponding average power of 1.2 W output at a pulse repetition rate of 10 Hz for 15 s in the MSP mode (pulse length 100 μs). Energy density was 9.04 J/cm^2. The tip diameter was 1.3 mm. The levels for air and water were 90 and 80 %, respectively.

In the two groups with laser etching, irradiation was manually performed in a single direction on a bonding area that was smoothly scanned only once. The tip–specimen distance was adjusted to 1 mm.

One investigator (S.A.) performed the etching procedure in the acid and Er:YAG laser etching groups, while another investigator (N.D.) performed the etching procedure in the femtosecond laser etching group.

After the etching procedures, one specimen from each group was selected for the observation of enamel surfaces under a scanning electron microscope (SEM). The enamel

surfaces were evaluated according to the enamel damage index (EDI) as follows [16]: grade 0, smooth surface without scratches, and perikymata may be visible; grade 1, acceptable surface with fine scattered scratches; grade 2, rough surface with numerous coarse scratches or slight grooves; and grade 3, surface with coarse scratches, wide grooves, and enamel damage visible to the naked eye.

Stainless steel, pre-adjusted, edgewise maxillary premolar brackets (0.22-in. slot; Mini Master Series Brackets, American Orthodontics, Sheboygan, WI, USA) were bonded with Transbond XT (3M Unitek, Monrovia, CA, USA) to the conditioned surfaces in all groups. Adhesive resin was applied to the bracket base, and each bracket was positioned onto a buccal enamel surface. All brackets were pressed firmly, and excess adhesive was removed with a sharp explorer. Then, the adhesive was light cured for 20 s. All bonding procedures were performed by one investigator (S.A.).

The average surface area of the bracket base was calculated as 11.21 mm^2 using a digital caliper (Absolute Digimatic; Mitutoyo, Miyazaki, Japan).

SBS testing

The bonded specimens were stored in artificial saliva and incubated at 37 °C for 24 h, followed by a thermocycling process (5000 cycles, 5–55 °C, Fig. 1).

SBS testing was performed using a universal testing machine (Shimadzu AG-X, Tokyo, Japan) at a speed of 0.5 mm/min until failure (Fig. 2). The specimens were oriented such that the buccal surfaces of the crowns were parallel to the long axis of the tooth. The force required for debonding was obtained in Newtons (N) and converted into megapascals (MPa) by dividing the value by the bracket base surface area.

$$\text{SBS (MPa)}$$
$$= \text{Debonding force (N)/Bracket base area} \left(\text{mm}^2\right); \ 1 \ \text{MPa}$$
$$= 1 \ \text{N/mm}^2$$

Failure mode analysis

The bracket bases from the three groups underwent surface morphological analyses using a stereomicroscope (SMZ 1000 Nikon; Nikon Corporation, Tokyo, Japan) to determine the adhesive remnant index (ARI) using the method proposed by Artun and Bergland. Calculations were based on the amount of adhesive left on the enamel surface, and scoring was as follows: 0, no adhesive remaining on the tooth surface; 1, less than 50 % adhesive remaining on the tooth; 2, more than 50 % adhesive remaining on the tooth; and 3, 100 % adhesive remaining on the tooth, with a distinct impression of the bracket mesh.

Fig. 1 The bonded specimens were stored in artificial saliva and incubated at 37 °C for 24 h, followed by a thermocycling process (5000 cycles, 5–55 °C)

For SEM analysis, the ceramic samples were first sputter-coated with gold–palladium particles (Cressington Sputter Coater 108Auto, Cressington MTM-20, Elektronen-Optik-Service, Dortmund, Germany) for 15 s to obtain a 90-Å-thick layer. The surfaces were then observed at ×19–×1000 magnification with a stereo-electron microscope (Evo LS10, Carl Zeiss, Oberkochen, Germany).

Statistical analysis

SBS data for the three groups were subjected to normality testing using the Kolmogorov–Smirnov test. Non-normal
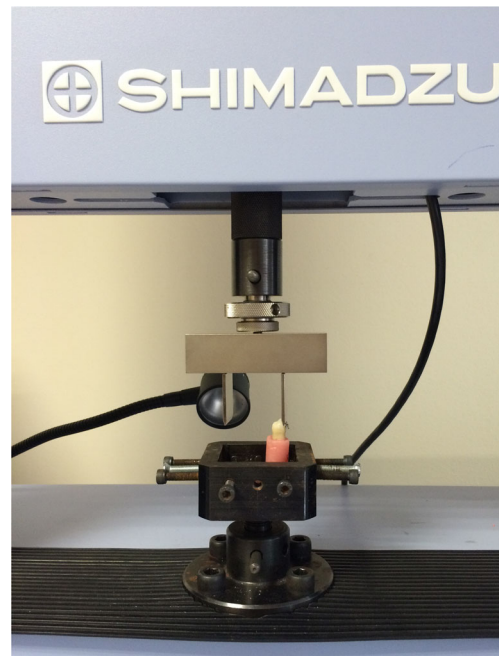

Fig. 2 SBS testing was performed using a universal testing machine (Shimadzu AG-X, Tokyo, Japan) at a speed of 0.5 mm/min until failure

distributions were observed; therefore, a nonparametric test (Kruskal–Wallis) was used to determine the significance of differences among groups. The Mann–Whitney U test was performed to determine differences among groups. The level of significance was set as $p < 0.05$. ARI scores were evaluated using chi-square tests.

Results

The SBS values for the three groups are shown in Table 1. There was no significant difference between the Er:YAG laser (7.49 ± 3.4 MPa) and acid etching groups (7.55 ± 2.8 MPa), whereas there was a significant difference between the femtosecond laser etching group (3.39 ± 1.2 MPa) and the other two groups ($p < 0.01$).

Bond failure modes are shown in Table 2. ARI scores were significantly different among the three groups ($p < 0.05$; Table 2). The acid etching groups showed a greater distribution of scores 2–3, whereas the two laser etching groups showed a greater distribution of scores 0–1.

SEM images obtained after the etching and debonding procedures are shown in Figs. 3, 4, and 5. No fractures or microcracks were observed in irradiated enamel from any group.

The Er:YAG laser and acid etching groups demonstrated coarse scratches, wide grooves, and enamel damage visible to the naked eye on SEM images obtained after etching (EDI grade 3; Fig. 3a, c). However, in the femtosecond laser etching group, an acceptable surface with fine, scattered scratches was observed (EDI grade 1; Fig. 3). No fractures or microcracks were observed in the etched enamel.

Figure 4 shows SEM images of tooth surfaces after debonding. The enamel–adhesive interface showed good micromechanical interaction with the enamel in the Er:YAG laser etching group (Fig. 4a). In the femtosecond laser etching group, a slight micromechanical interaction was observed at the interface of the enamel and the bonding side, with some remaining resin (Fig. 4b). In the acid etching group, numerous and continuous enamel tags were observed (Fig. 4c).

Table 1 Descriptive statistics of shear bond strengths in megapascals and comparison of the groups

	Number	Min	Max	Mean ± SD	Comparison
Group MSP	17	21.6	172.6	7.49 ± 3.4	A
Group FTS	17	11.4	62.5	3.39 ± 1.2	B
Group control	17	20.0	147.6	7.55 ± 2.8	A

Table 2 Comparison of adhesive remnant indices of groups

	Number	0	1	2	3
Group MSP	17	7	10	0	0
Group FTS	17	3	12	1	1
Group C	17	1	4	7	5

ARI scores: 1 = less than half of the adhesive left on the tooth surface, 2 = half of adhesive or more left on the toth surface, 3 = all adhesive left on the tooth surface

Discussion

In the present study, Er:YAG and femtosecond laser etching techniques were compared with the conventional acid etching technique with regard to SBS values for bonded brackets, ARI scores, and SEM images. The results demonstrated differences among the three groups; therefore, the null hypothesis was rejected.

The bond strength is affected by temperature variations in the oral cavity. Although testing is difficult, it is important to determine whether these variations introduce stresses in the adhesive that may influence its bonding strength. The most commonly used artificial aging technique is thermocycling [17]. Accordingly, we used a thermocycling procedure to simulate conditions in the oral cavity before SBS testing in this in vitro study.

Acid etching was used in the control group in the present study. It is the most popular etching technique for resin tags occurring on the enamel surface [13, 14]. However, the possibility of enamel decalcification is an important limitation and the main cause of dental caries. Laser technology has been widely used in dental practice since its advent, with studies demonstrating it to be safe for use in bracket bonding procedures in orthodontic practice [14].

Er:YAG lasers are effective for hard dental tissue. Several studies have investigated SBS values achieved with Er:YAG lasers, although the reported results are conflicting [9, 12, 13]. In recent years, the femtosecond laser has come to be used in various fields of medicine and dentistry.

Clinically acceptable bond strength values in orthodontics range from 6 to 8 MPa [18, 19]. In the present study, Er:YAG laser and acid etching resulted in adequate SBS values and femtosecond laser etching resulted in the lowest SBS values that were not adequate for clinical application among the three groups. Sagir et al. [13] similarly reported no significant difference in SBS values between Er:YAG laser and acid etching. However, overall results remain conflicting, with some previous studies [12, 20] documenting lower bond strengths with Er:YAG laser etching than with conventional acid etching and some [21, 22] documenting comparable or even stronger bond strength values.
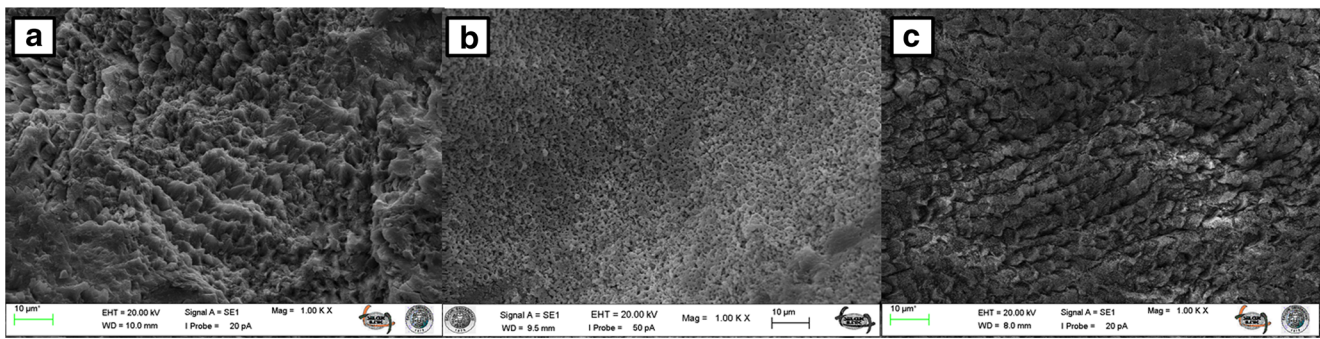
Fig. 3 The Er:YAG laser and acid etching groups demonstrated coarse scratches (**a**), wide grooves (**b**), and enamel damage (**c**) visible to the naked eye on SEM images obtained after etching (EDI grade 3)

A few studies have reported the enamel etching results for femtosecond lasers and observed higher SBS values that were not consistent with those obtained in the current study [9, 10, 14]. Kabas et al. [14] reported that a Yb:glass femtosecond laser with an output setting of 120 mW resulted in SBS results that were comparable with those obtained with conventional acid etching; 80 mW did not achieve the minimal value. In the present study, an output setting of 0.4 W was used, which was lower than that used by Kabas et al. [14]. The discrepancy in findings could also have resulted from the use of ceramic brackets instead of metal brackets, which have higher SBS values [23]. Moreover, bovine teeth were used in the previous study, and it remains unclear whether data obtained from bovine teeth can be applied to human teeth for several reasons, including the origin of enamel [18, 24].

Lorenzo et al. [9] compared acid etching, Er:YAG laser etching, and femtosecond laser etching and reported that the SBS value obtained with femtosecond laser etching (22.9 MPa) was higher than that obtained with Er:YAG laser etching (7.8 MPa). All SBS values in their study were nearly two to three times higher than those obtained in the current study. Moreover, femtosecond laser etching and conventional acid etching resulted in equivalent SBS values. This difference in results can be primarily attributed to the thermocycling procedure applied to all samples in the present study. We

applied thermocycling before SBS testing to simulate intraoral thermal changes and water absorption that occurs during prolonged orthodontic treatments [13, 25]. Another reason for the different results can be the differences in study protocols. Previous investigations have demonstrated different power and irradiation settings, and the distance between the laser tip and the enamel surface affects SBS values [8, 13]. Speed, the repetition number (passes through the same point), the laser pulse repetition rate (kHz), and the laser pulse power influence the depth and width of craters on the enamel surface [11].

Even if the debonding force is the most important parameter for comparing different etching methods, the overall time spent on bracket bonding is also crucial when determining the materials and procedure for conditioning the enamel surface [14]. The effects of laser applications are dependent on the exposure time [14]. Previous studies showed that femtosecond etching took remarkably longer than acid etching. A denser femtosecond laser pattern has been reported to improve the bracket bonding strength on enamel surfaces [10]. However, different surface patterns with SBS values higher than those observed in the current study require 15–120 min/surface for bracket etching [10]. These results indicate that full-mouth etching can take approximately 6 h, which is unacceptable in clinical practice [10]. In this regard, the difference in our
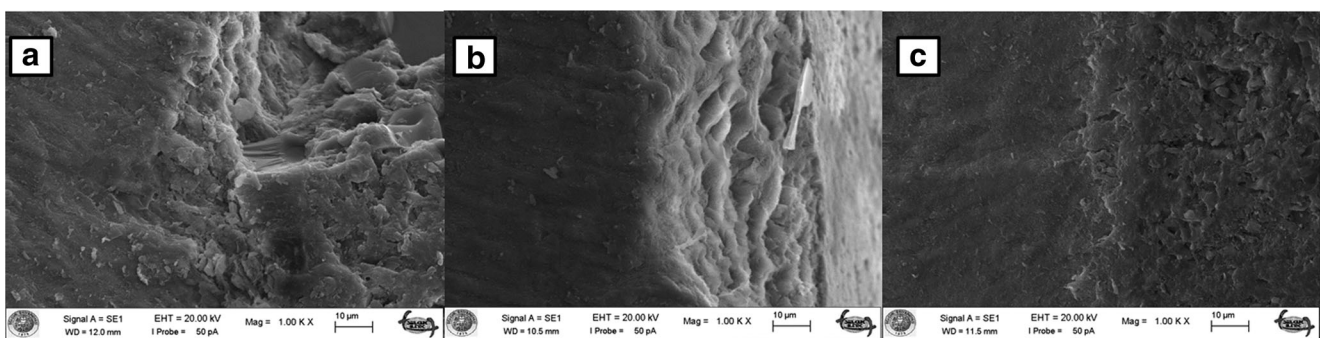

Fig. 4 SEM images of tooth surfaces after debonding. **a** The enamel–adhesive interface showed good micromechanical interaction with the enamel in the Er:YAG laser etching group. **b** In the femtosecond laser etching group, a slight micromechanical interaction was observed at the interface of the enamel and the bonding side, with some remaining resin. **c** In the acid etching group, numerous and continuous enamel tags were observed
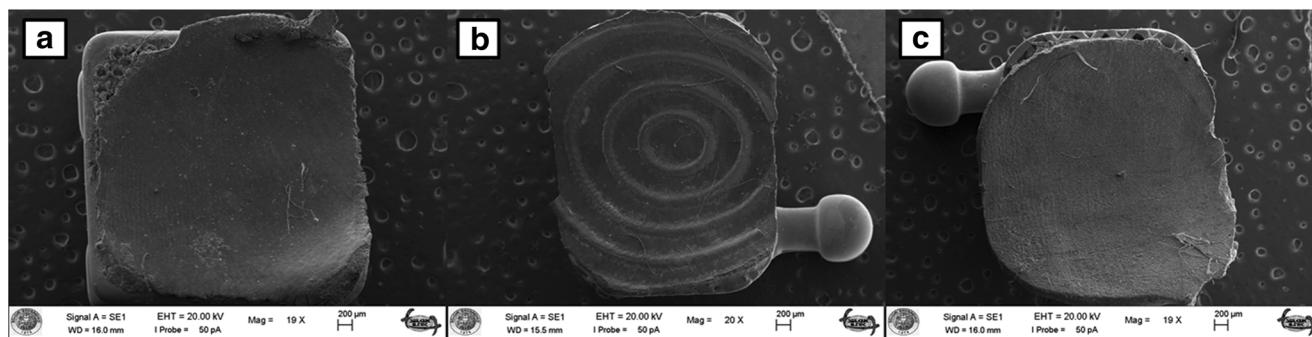
Fig. 5 SEM images obtained after the etching and debonding procedures. **a** ErYAg, **b** Femtosecond, **c** Acid

findings can be attributed to the use of irradiation on the bonding area, which was smoothly scanned only once, thus corresponding to a feasible duration for laser etching in simulated clinical practice.

SEM images were acquired for visual evaluations among the three groups in the present study. Irregular and rough surfaces were observed in the Er:YAG laser etching group, whereas the acid etching group demonstrated more regular surfaces. After polymerization, craters and microcracks were observed in the Er:YAG laser etching group. Micromechanical interlocking of resin tags within the acid-etched enamel surfaces, which provides the best achievable adhesion, was observed in the acid etching group, similar to the observations in previous studies [22, 26].

In the femtosecond laser etching group, a uniform surface with micropores was observed. However, the surface was clearly less rough than that in the acid and Er:YAG laser etching groups. The depth of the micropores was not adequate to generate micromechanical retention, which allows for the incorporation of small resin tags within the enamel surface (Fig. 4b).

Good adhesion requires the exposure of enamel rods and collagen fibrils [27]. However, in the femtosecond laser etching group in the present study, the enamel surface mostly behaved like an unaltered surface. The obtained SBS values and SEM findings were in agreement with each other. Nonetheless, SEM evaluations were based on single specimens from each group; therefore, the results should be interpreted with caution. Further studies with larger sample sizes are necessary to determine the extent of the surface morphology and obtain quantitative data.

ARI scores evaluate the presence of remaining resin on the enamel and classify the amount according to an objective scale. It is desirable to have no resin attached to the enamel surface [14]. In the present study, the enamel–adhesive interface was the most common bond failure site in both groups with laser etching. The ARI scores obtained in this study were consistent with those obtained in previous studies that used femtosecond and Er:YAG lasers [8, 13, 14]. Furthermore, they demonstrated that laser etching results in a clean enamel surface, which decreases the time required to clean teeth after debonding [13].

The ARI scores in the acid etching group primarily represented failure at the bracket–adhesive interface. Failure at the bracket–adhesive interface is considered safer because enamel fracture and crazing have been reported during bracket debonding [26]. Conventional acid etching may increase the depth of microretention, resulting in deeper and more retentive resin tags and the presence of more residual adhesive on the enamel surface [9].

ARI scores were significantly different among the three groups in the present study. Although the preferred site of failure is controversial, laser systems have an advantage in terms of less chairside time, while acid etching is more desirable to prevent enamel fracture. Nevertheless, surfaces exhibiting large amounts of resin after debonding are at risk of iatrogenic damage with regard to increased enamel loss during cleaning after the debonding procedure [10].

This study was conducted as an in vitro study. In vivo testing in controlled trials is the best way to test the effectiveness of a bonding system and observe any detrimental effects to the enamel. However, in vitro studies possibly allow for more standardized testing procedures for a specific etching system [18]. As a consequence, further studies are needed with different power settings to increase SBS values and decrease the time required for routine enamel conditioning procedures under actual clinical conditions.

Conclusions

The results of the present study suggest that Er:YAG laser etching and conventional acid etching are superior to femtosecond laser etching in terms of bracket bond strength and enamel demineralization. The sole use of femtosecond lasers for enamel conditioning may be inadequate, because of the resulting low SBS values and insufficient micromechanical retention.

To overcome the limitations of this in vitro study, further studies with different power settings to increase SBS values and decrease the time required for enamel surface conditioning before bracket bonding in routine clinical setting are necessary.

Compliance with ethical standards

Conflict of Interest   The authors declare that they have no conflict of interest.

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