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Abstract. The aim is to investigate femtosecond laser ablation as an alternative method for enamel etching used before bonding orthodontic brackets. A focused laser beam is scanned over enamel within the area of bonding in a saw tooth pattern with a varying number of lines. After patterning, ceramic brackets are bonded and bonding quality of the proposed technique is measured by a universal testing machine. The results are compared to the conventional acid etching method. Results show that bonding strength is a function of laser average power and the density of the ablated lines. Intrapulpal temperature changes are also recorded and observed minimal effects are observed. Enamel surface of the samples is investigated microscopically and no signs of damage or cracking are observed. In conclusion, femtosecond laser exposure on enamel surface yields controllable patterns that provide efficient bonding strength with less removal of dental tissue than conventional acid-etching technique. © 2013 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: 10.1117/1.JBO.18.9.098003]

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1 Introduction

The practice of orthodontics prevents and corrects irregularities of the teeth and oral tissues to create a more esthetic and functional dental system. Brackets are one of the fundamental parts in orthodontics applications. Brackets are fixed on enamel, so they must be stable during treatment.^{1,2}

After Buonocore³ introduced the technique of acid etching of the enamel surface, Newman⁴ developed the direct bonding method for the adherence of brackets after applying composite resins on the acid-etched surfaces. Acid etching is the selective dissolution of enamel that causes microporosities, resulting in bonding via mechanical retention.⁵

The classical bonding procedure consists of cleaning the hard tissue surface, etching the enamel with acid, rinsing, and drying. The micromechanical interlocking system is achieved by the penetration of the resin tags into the microporous substrate.⁶ The bonding strength of the brackets is enhanced by mechanical retention.^{5,6} An enamel loss of a depth of 10 μm to 50 μm results from acid etching.^{7,8}

The primary disadvantage of acid etching is the possibility of decalcification, which renders the enamel susceptible to caries attacks. The discoloration of enamel due to resin tags is another handicap of this method. These drawbacks motivated the studies for novel techniques or methods. The laser-etching technique is one of the most promising alternative applications under investigation.

Various lasers have been used for the etching of enamel and dentin since the first application of lasers in the dental field.⁹⁻²⁶ Laser irradiation on the enamel surface causes physical changes such as melting and recrystallization. Advantageously, it yields a

comparable surface roughness to acid etching in terms of structural changes.¹¹

The effects of laser applications are dependent on the laser wavelength, power, mode of operation (pulsed, continuous wave, femtosecond pulses), and exposure time. As there are several studies regarding the optimum laser parameters to improve etching applications, irradiation was proposed as a feasible method to etch surfaces of hard dental tissue.¹⁵⁻¹⁷

Laser etching reduces the risk of demineralization and enamel damage while decreasing the required debonding force.^{16,17} In addition, laser etching requires less time than the conventional acid-etching procedure.⁸⁻¹⁷ Previous studies investigated the effects of the laser parameters as well as the combination of laser applications with acid-etching procedures.¹⁵

Despite these advantages, undesired thermal side effects and a lack of efficiency were reported as the disadvantages of lasers in most cases. It has been reported that irradiation of dental tissue can result in thermal damage,²⁷ and the sharp increase in temperature during laser etching may form cracks and therefore subsequent bonding failures.¹⁷

Although some studies on the optimum laser parameters showed minimal pulpal inflammatory responses via histological studies,¹⁵ the role of microcracks in laser etching is still a controversial subject. Although they contribute to mechanical retention, cracks are accepted as the starting points for new carious attacks. Therefore, they must be avoided for the long-term success of dental treatments.

Previous studies aimed to overcome these negative side effects by controlling the parameters. The advantages and limitations of using ultrashort laser pulses with either picosecond or femtosecond durations were discussed as a viable solution. The most effective improvement of super-pulse lasers exhibited a reduction in the formation of microcracks.¹⁵ Moreover, the

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precise removal of dental hard tissue with minimal or no thermal damage was accomplished by the application of ultrashort laser pulses.²⁸ The major advantage of these brief pulse durations was the ability to produce precise cavities without significant thermal side effects. The cavities opened by an ultrashort laser featured a precise geometry and the edges were clean and sharp, without thermal or mechanical damage.²⁸

Recently, Lorenzo et al. published a paper on enamel etching via femtosecond lasers for metal brackets and revealed that a femtosecond laser without the supporting acid application could have satisfying results.²³

The aim of our study was to investigate the etching performance of a 1030-nm femtosecond laser. The focused laser beam scanned the enamel surface at a constant speed with a varying number of ablation lines. Ceramic brackets were bonded and the teeth were kept in incubator for an adequate amount of time to ensure resin polymerization. A universal testing machine, which was used for debonding the brackets, measured the bond strength. The bonding strength of the ceramic brackets was compared to that of the conventional acid-etching technique. A scanning electron microscope (SEM) was used to examine the microcracks and morphological changes that occurred on the enamel surface. Temperature measurements were also recorded during the laser exposure.

This present study features the first use of a 1030-nm femtosecond laser for the etching of enamel to bond orthodontic brackets. Ceramic brackets were chosen because of their aesthetic priority, which enjoy popularity despite the fact that they require more micromechanical retention for bonding as compared to that of the metal brackets.

2 Materials and Methods

2.1 Tooth Sample Preparation

Bovine enamel is accepted as a suitable alternative enamel to human enamel for bonding tests;^{13,16} therefore bovine teeth were used in this study because of their higher availability and hygiene. After extraction, the teeth were scaled off to remove calculus and soft tissue debris and then they were rinsed with water. The bovine teeth were embedded in gypsum blocks to model the labial surface of the crown and they were positioned as parallel as possible to the vertical axis of the block. Cavities were opened on the lingual surface of the teeth using a round diamond bur and then they were filled with silicon paste.

2.2 Bonding Procedure

Bis-phenol-A-glycidyl methacrylate adhesive resin (3M) was selected because of its widespread use in clinical procedures. The steps of the bonding procedure for direct bracket bonding on lingual surfaces were carried out as per the manufacturers' recommendations: cleaning, etching, sealing, and bonding. In our study, two different etching procedures were applied for two different types of etching groups: conventional acid etching and laser etching.

In the acid-etching group, each cleaned and polished enamel surface was etched for 30 s with acid. After rinsing with a syringe and then being dried, a chalky and frosty white appearance was observed on the enamel surface, indicating proper etching. After the etched area was completely dry and frosty white, a thin layer of bonding agent (sealant) was painted over this surface and the bracket base. After the bonding

procedure, the samples were placed in a beaker with an isotonic solution and stored at 100% humidity and 37°C for more than 2 days to ensure polymerization of the composite.

2.3 Laser Patterning Setup

To examine the effectiveness of femtosecond laser pulses for enamel etching, a Yb:Glass femtosecond laser operating at a wavelength of 1030 nm was employed (s-pulse, Amplitude Systemés, France). The repetition rates of these pulses were 1 kHz, and the pulse duration was 550 fs.

The experimental setup was composed of a Yb:Glass femtosecond laser system, a half waveplate, a polarizer, a galvo system, a signal generator, and a lens (Fig. 1). A Gaussian laser beam was transmitted through a waveplate and a polarizer to adjust the irradiation power. The transmitted light was focused on the fixed sample surface using a spherical lens with a focal length of 100 mm. The two-axis galvo-scan system (GVS012/M, Thorlabs, New Jersey) was used to scan the laser beam on the surface at a constant speed of 1 mm/s. For all samples, the scanned area was $3.5 \times 3.5 \text{ mm}^2$, which was equal to the size of the bracket base. A signal generator controlled the mirrors in order to obtain a saw tooth pattern on the sample surface with the laser beam.

For each value of power, three different groups with a varying number of scanning lines were evaluated. The scanning groups were composed of 20, 30, and 40 lines within the defined area. Ten samples were measured in each group.

2.4 Ablation Profile and Surface Measurements

The cross-sectional profiles of the laser-ablated sites were examined using a surface profilometer (Veeco, Dektak 150). The depth and width of the lines were measured and the average value was calculated for each experimental group.

The ablated surfaces were investigated via optical microscopy (Nikon, Eclipse, LV150L). Sample microscope images for the three different line densities are shown in Fig. 2. An SEM imaged the surface in more detail, enabling the investigation of cracks.

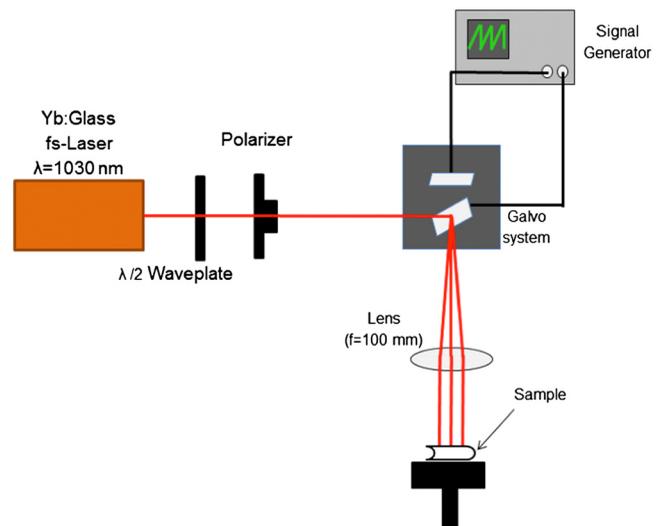


Fig. 1 Schematic setup for Fs-laser etching experimental procedure.

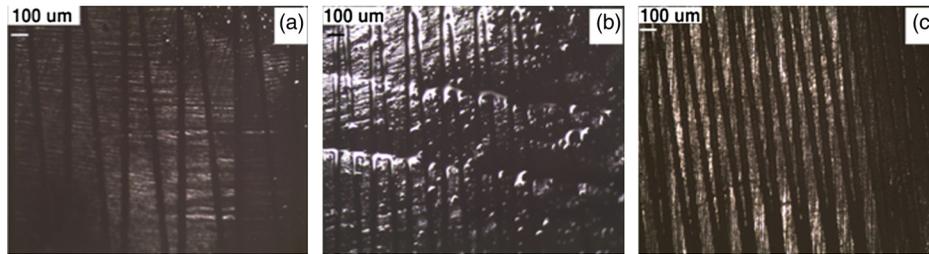


Fig. 2 Optical microscope images of fs-laser patterned teeth surfaces at 100 mW average laser power for (a) 20, (b) 30, and (c) 40 lines.

2.5 Temperature Measurements

A K-type thermocouple (OMEGA, OM-CP-0CTTEMP, UK) was used in the temperature measurement system to measure intrapulpal thermal changes during irradiation. A computer read, collected, and recorded the data from the K-type thermocouple.

2.6 Debonding Force Measurements

A universal testing machine (Lloyd, LF Plus, UK) measured the shear bond strength of each specimen during debonding. It consisted of an upper moving section and a rigid base section. A steel shearing blade was also mounted on the upper part of the machine. The base section was prepared as a testing frame. To debond the brackets from the teeth, the gypsum blocks were placed in this testing frame. A moving arm directly transmitted the force to the ceramic bracket; the direction of the force was perpendicular to the bracket. Every specimen was tested in the same manner to compare the forces among the four groups. In addition to this, the computer connected to the testing machine controlled the machine as well as read, collected, and recorded the data.

2.7 Adhesive Remnant Index (ARI) Scores

The adhesive remnant index (ARI) system was used to evaluate the amount of adhesive left on the tooth after debracketing. Among several defined ARI scores, a scoring system of 0 to 3 was selected for this study.^{26,29,30} The criteria for scoring are as follows:

- Score 0 = no adhesive left on the tooth;
- Score 1 = less than half of the adhesive left on the tooth;
- Score 2 = more than half of the adhesive left on the tooth;
- Score 3 = all adhesive left on the tooth with a distinct impression of the bracket mesh.

Environmental scanning electron microscope (ESEM) observation is an effective method for determining the minimal differences throughout the etched enamel area and precisely comparing the procedures. Therefore, it is widely used in examinations on irradiation.

3 Results

In this study, a Yb:Glass femtosecond laser system etched the enamel surface. Ten groups were studied with irradiation powers of 80, 100, and 120 mW and etching patterns of 20, 30, and 40 lines. Because the intrapulpal temperature varies during irradiation, the ablation depths and the debonding forces were measured for each sample. The ARI scores were examined for the

existing groups. In addition to this, SEM observations of 80-, 100-, 120-, and 200-mW lased samples were imaged (Table 1).

3.1 Temperature Measurements

The variation of intrapulpal temperature for all laser groups was measured in the range of 0.7°C to 1.6°C, which was significantly under the threshold value reported in the literature.¹⁹

3.2 Ablation Depths

Grooves, which were conically shaped, on the enamel formed via laser ablation for all three laser powers. Although the samples exposed to an irradiation of 80 mW had a higher mean value, all three laser powers exhibited similar ablation depths. There was no significant difference among the measurements taken from the different ablated areas according to the analysis of variance (ANOVA). In addition to this, there was no significant difference in chemically etched and laser-etched samples Fig. 3.

3.3 Debonding Forces

Two different enamel etching methods, conventional acid etching and laser etching, were compared in this study. According to

Table 1 Each experimental group and the examinations done on samples were shown below.

Groups	Observations
Acid-etching group (control group)	<ul style="list-style-type: none"> • Debonding force measurements • ARI score examinations • SEM observations
Laser-etching groups	<ul style="list-style-type: none"> • Debonding force measurements • ARI score examinations • Temperature measurements • SEM observations • SEM observations
80 mW	<ul style="list-style-type: none"> 20 lines 30 lines
100 mW	<ul style="list-style-type: none"> 40 lines 20 lines 30 lines 40 lines
120 mW	<ul style="list-style-type: none"> 20 lines 30 lines 40 lines
200 mW	<ul style="list-style-type: none"> 20 lines

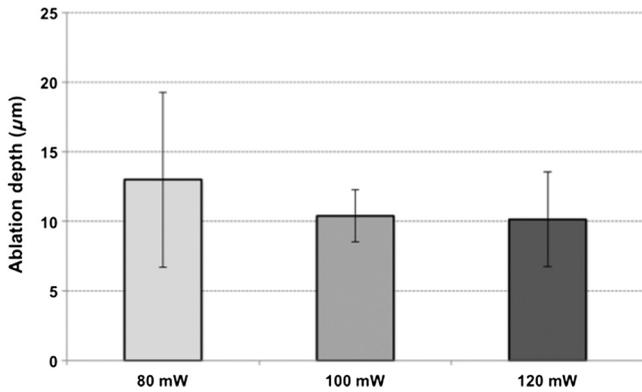


Fig. 3 The depths of ablated areas on enamel were measured by the surface profilometer and graphed as above. There was no significant difference between the measurements belonging to ablated areas according to the ANOVA.

these results, a significant difference was observed between the conventional etching and laser techniques (Student *t* test, $p < 0.05$) (Fig. 4).

The laser power and the number of lines were the two variable parameters examined in the laser group. There was a significant difference among the debonding forces of the lased samples (as determined by the ANOVA method). Nevertheless, neither the change in the laser output nor the variation in ablation areas resulted in a significant difference individually. Thus, a substantial difference was only possible when both of these parameters were varied simultaneously. Furthermore, the Tukey test indicated that the laser power affected the debonding; it also indicated that altering the number of ablation lines did not affect the debonding. The difference among the groups with varying number of lines with the same power was less for the 80-mW group than for the other laser groups.

The debonding forces were between 6.1 and 21.2 N for the conventional method. All three groups irradiated at 80 mW showed a lower debonding force than the minimal value of the acid-etched group. The 100 mW/40 lines group and

120 mW/30 lines and 120 mW/40 lines groups exhibited comparable debonding forces to the control group. However, the 100 mW/40 lines group and 120 mW/20 lines and 120 mW/40 lines groups exceeded the maximum debonding force value of the acid-etched group. The debonding force of the 120 mW/20 lines group was between the minimum and maximum values and ideally represented the results of the conventional acid etching. As a result, this laser group possessed the most optimal results among all the groups.

3.4 Optical Microscope and ESEM Observations

Examinations completed with an optical microscope showed no evidence of thermal damage, such as surface cracking or carbonization, in any of the experimental groups. Additionally, precise ablation and sharp edges of the ablated areas were observed.

The irradiation at powers of 80, 100, 120, and 200 mW with the same number of lines were observed by an ESEM (FEI-Philips XL30 ESEM-FEG, Holland). The imaging was completed at magnifications of 40×, 100×, 1000×, 2000×, 4000×, 5000×, and 10,000× (Fig. 5).

The conventional etching method clearly occurs via type II etching.³¹ As there was no selective melting location throughout the lased surface areas, the irradiation was determined to be due to type III etching. No fracture or microcracks were observed on the irradiated enamel.

The comparison of the SEM images showed that the 100-mW lased enamel was noticeably different from the other laser groups. Especially above the 4000× magnification, the radius of the microexplosions was briefly larger for the 100-mW irradiated group. The width of the ablated groove proportionally increased with the lasing power. This observation is more evident in the 120- and 200-mW irradiated samples.

3.5 ARI Scores

According to the ARI scale of 0 to 3 used in this study, the ARI enamel results of the laser groups most frequently scored 0 (93%); the remainder scored 1 (7%). No ARI enamel scores

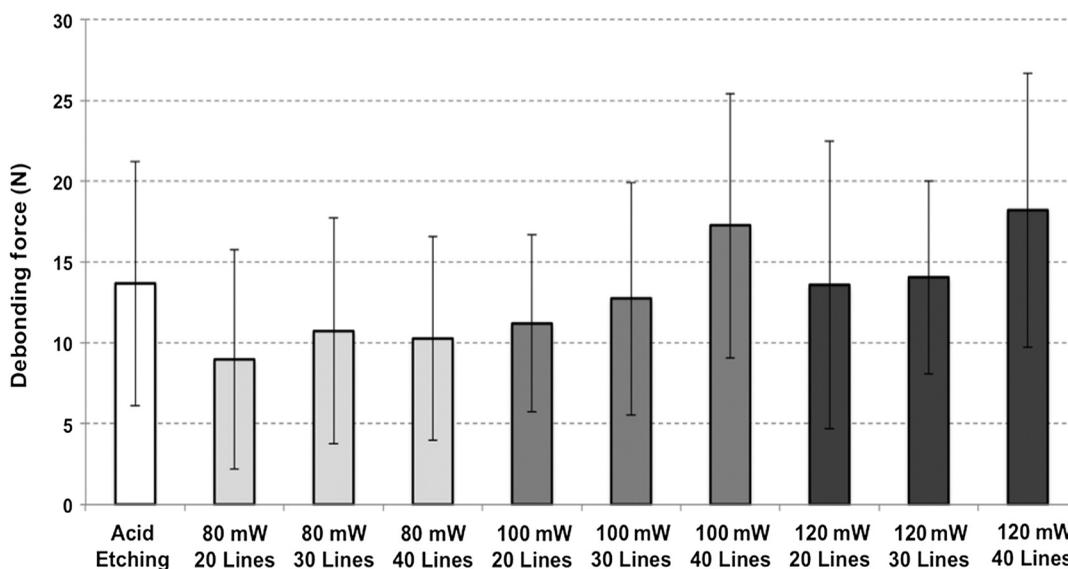


Fig. 4 The universal testing machine measured the shear bond strength of each specimen during debonding. Thus debonding forces showed a significant difference between conventional etching technique and laser groups (Student *t* test, $p < 0.05$). Besides, a significant difference was observed in debonding forces among lased groups according to the ANOVA. Tukey test showed that varying laser power caused significance difference while difference in the number of ablation lines did not cause any difference.

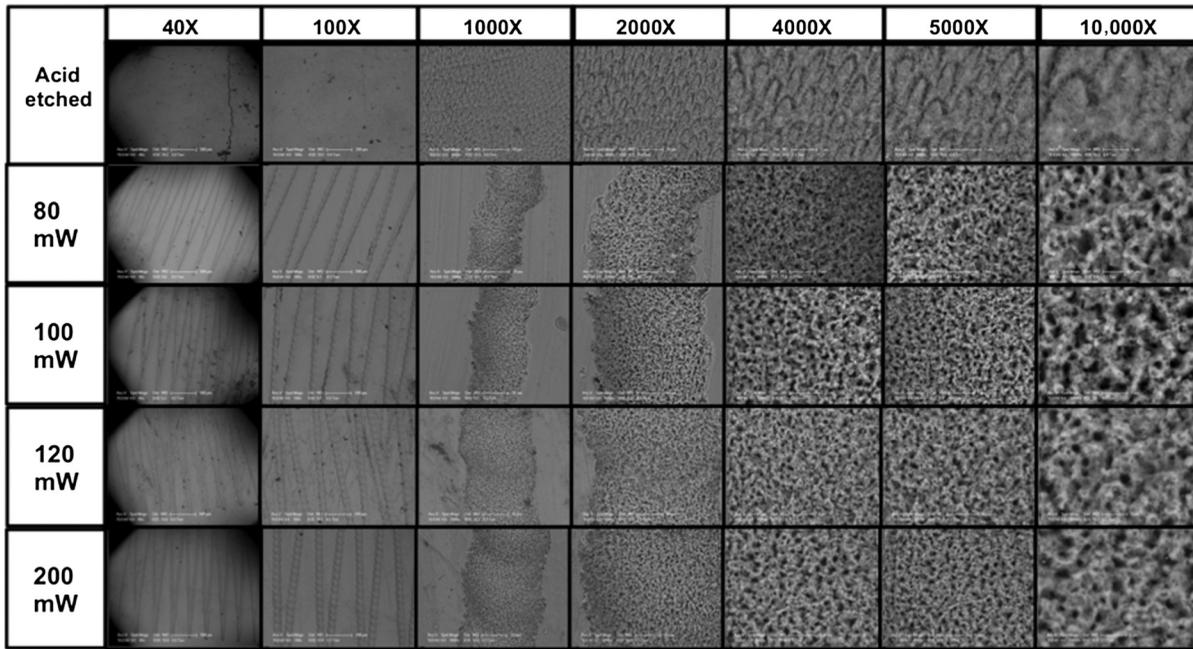


Fig. 5 SEM examinations were made for observing the irradiated enamel by an environmental scanning electron microscope. The recorded photos were grouped according to the laser power and magnification types.

of 2 or 3 were present in the irradiated groups. In addition to this, the acid-etched group had only 1 out of 11 samples with a score of 2; the remainder of the samples had a clean enamel surface with a score of 0.

4 Discussion

Although acid etching is the most popular enamel etching technique in clinics today, the possibility of enamel decalcification is a serious drawback. Laser etching studies aim to propose a safer technique for the practice of bracket bonding. However, the usage of lasers creates two important problems. Depending on the laser used, a possible temperature increase may harm dental tissue or result in pain for the patient. Second, mechanical damage may occur in the form of microcracks on the enamel surface. Laser etching studies concentrate on these problems, and various laser parameters and modes of operation have been tested.¹⁵ Particularly, ultrashort pulse lasers are suggested to limit the negative side effects during irradiation.³²

A 1030-nm Yb:Glass fiber femtosecond laser was selected for this study to determine the optimal laser etching conditions. Ten laser groups with varied irradiation powers and etching patterns were examined and compared to the conventional acid etching method. The results were evaluated in terms of the parameters: (1) intrapulpal temperature changes during irradiation were recorded; (2) ablation depths of lines on the enamel surface were measured; (3) debonding forces after treatment were measured; (4) ARI scores were determined; and (5) enamel surfaces were examined via SEM observations to detect microcracks.

4.1 Temperature Measurements

Thermal damage is always a risk for laser applications. The choice of laser wavelength and the mode of operation (continuous or pulsed) can minimize the negative thermal effects. The

optical properties of dental tissue change with respect to the laser wavelength. Poorly absorbed wavelengths can result in more thermal damage to the surrounding tissue. Contrary to Nd:YAG and diode lasers, Er:YAG and Er:YSGG lasers are well absorbed by water and are accepted as convenient lasers for minimizing thermal damage. However, the thermomechanical ablation from these lasers can generate microcracks on enamel because of the sudden increases in local temperature.²⁴

In the present study, all the groups that were etched with a 1030-nm Yb:Glass femtosecond laser showed an acceptable intrapulpal temperature increase during lasing. Our results were found to be similar to that of a previous study¹⁵ and the temperature increase we recorded was below the threshold²⁷ for probable histological changes in pulp tissue.

4.2 Ablation Depths

In the present study, we created ablation lines on the enamel surface via femtosecond laser scanning. Deeper ablation lines ensure better resin penetration, but dental material removal should be limited. The measurements of the debonding forces yielded data for determining optimal ablation depths.

The possible enamel loss depth during etching procedures varies between 10 μm and 50 μm .^{7,8} The conventional acid-etching technique also results in the removal of a similar amount of enamel.³³ Our study agreed with other laser etching studies; there was no significant difference in roughness between our laser-etched surfaces and conventionally etched surfaces.¹⁷ In addition, none of our samples in the irradiated groups exceeded the possible etching depths obtained via the acid-etching procedure.

Different laser powers were applied, but no significant difference was observed in the depths of the ablations on enamel. However, an increase in power should result in deeper ablations. The melted enamel in the groups with a higher laser power may have filled the ablated area, and thus, decreased their overall

ablation depth. Nevertheless, the ablated volume was found to be acceptable with reference to the acid-etching removal of enamel.

4.3 Debonding Forces

The debonding force is the most important parameter in comparing the different etching methods. In this study, debonding forces similar to the forces measured for the acid-etched enamel in literature are accepted. Some studies reported that acid-etched teeth had a significantly higher bond strength than laser-etched teeth,^{13,15,16} whereas others demonstrated that laser etching could result in a bond strength comparable to or even stronger than acid etching.¹⁷ In addition, some studies revealed that a laser could be effective if a high lasing power was applied,⁸ however, this may cause adverse thermal effects.

In the present study, laser etching did not provide the ideal enamel topography, but the debonding force values in this study proved that type III etching facilitated adequate penetration of the fluid adhesive components into the irregularities. There was no significant difference in the debonding forces between the conventional etching technique and the laser method.

After introducing the mechanism of pulsed laser ablation in 2003,¹⁵ obtaining an adequate bonding strength during treatment without thermal side effects became feasible.

An ultrashort super-pulsed laser was used in our study, and there were two different parameters compared in our laser groups. The density of the ablation lines and the laser powers were varied and analyzed in this study. The results showed that a significant difference could only be observed if both of the parameters were simultaneously varied.

Although previous studies defended that only the combination of laser etching and chemical etching could be capable of approximating the bond strength produced by that of chemical etching alone, a 1030-nm femtosecond laser was able to provide a comparably adequate debonding force.¹⁵

Similar to Lorenzo et al., our debonding forces obtained from a femtosecond laser were comparable to the values of the conventional etching method. In contrast to some negative results using other types of lasers in the literature, irradiation with a 1030-nm laser successfully provided an adequate bonding strength, as in the conventional method.²³

By analyzing the maximum and minimum points of the bonding strength values, it is possible to briefly extrapolate the differences among the groups. Values lower than the minimum or higher than the maximum debonding forces in the conventional method are not preferable. Although all three 80-mW groups were substantially under the maximal conventional value, there was no significant difference with the acid-etched group.

The success of laser treatment is directly related to the period that the ceramic bracket is able to stably remain on the tooth. The system must maintain its strength until the end of the treatment; therefore, comparing the minimal values for the bond strength is more meaningful in debonding force.

Although there is reported minimum value for the bond strength in the literature, the variety among bonding resins and ceramic brackets in the market complicated the comparison of the debonding forces in other studies.

Another important issue is the dry surface maintained via irradiation on enamel. Drummond et al. proposed that laser etching lowers the water content of teeth.¹⁶ They reported the wetting properties of this acetone-based sealant system were reduced with less moisture on the surface, and thus, sealant

penetration into the enamel surface undercuts is limited. As a result, reduced bond strengths were observed in the related studies.

Thus, regardless of the other studies' bonding strength results, the debonding forces were only compared to our control group values. Among the irradiated groups, all 80 mW groups were under the obtained minimal value in the previous method. The 100-mW irradiated group with 40 ablated lines was able to exceed the acceptable threshold. Additionally, in the 120-mW lased group, the ablated subgroups with 30 and 40 lines provided adequate minimal debonding forces. Although these three popular groups were accepted as optimal, the 120-mW lased group with 30 ablated lines was determined to be the optimum group according to the bond strength measurements.

4.4 ARI Scores

The ARI evaluates the presence of remaining resin on the enamel and classifies this amount according to an objective scale. It is desirable to have no attached resin on the enamel surface.

Although the debonding forces of the laser-ablated and acid-etched groups were somewhat equivalent in this study, the bond failure sites of the laser-etched treatment had a higher percentage of remnants on the bracket–resin interface. With reference to the ARI, this study had parallel results to the previous studies.²³ The ARI scores in this study demonstrated that our methods resulted in a cleaner enamel surface than the other femtosecond laser studies.^{22,23}

4.5 Optical Microscope and ESEM Observations

An enamel etching pattern is ideally characterized by a deep, uniform area of demineralization. However, laser irradiation generates uneven, heterogeneous surface characteristics with microcracks.^{9,17}

As mentioned above, the laser hard tissue interaction depends on the wavelength and the irradiation energy. The Nd:YAG laser is known for producing microcracks and fissures as a result of its thermal effects. Additionally, studies on popular hard tissue ablation lasers, Er:YAG and CO₂ lasers, also reported flaky structures and fine cracks observed on the enamel surface after irradiation.^{9,12,17}

Previous studies posited that using ultrashort pulsed lasers decreased the formation of microcracks on enamel. Our study agrees with this research on ultrashort pulse and femtosecond lasers; no debris, cracks, or fissures were observed via an optical microscope after irradiation with a 1030-nm Yb:Glass laser with the optimum parameters in this study.

Four laser power groups and the conventional acid-etched group were examined via ESEM observations. The conventionally etched surface exhibited type II etching whereas all irradiated groups exhibited type III etched pattern.

The ablation procedure resulted in the formation of a conical structure. In agreement with other femtosecond laser studies, precise laser ablation of enamel with well defined and clean edges was achieved in this study.

No difference among ablation depths of varying laser powers existed, as determined via profilometer measurements. Comparing ESEM images corroborated the initial measurements and showed that the radii of the microexplosions were the largest for the 100-mW groups. Furthermore, Bor Shiunn et al.¹⁷ published on the melted bubbles, craters, cracking,

and micropores in the enamel. Feurestein emphasized that the higher energy lasers exhibited features of melting.¹² As a result of these examinations and the previous studies, the present research defends that the equal ablation depths with respect to the increasing irradiation power were due to the melting of the enamel tissue. Additionally, the width of the ablated line proportionally increased with increasing lasing power.

In addition to all these advantages, our results agreed with Bor Shiunn et al., which mentioned that laser etching consumed less time than the conventional methods. Although it takes more than a minute for each tooth in an ideal-etching procedure that includes several steps, laser etching takes less than half the time of the conventional method with only a single step.

Another advantage the laser method features is with regards to controlling the area. In agreement with Øilo et al., this study supported that the bond strength is related to the size of the bonding area, so it is important to control this area.³⁴ In the present study, the size of the laser-etched area perfectly corresponded to the bracket base area. The laser method also generated a much smaller controllable ablated area than the chemical method.

5 Conclusion

Laser etching is a method increasing in recognition, but the improper use of light may cause thermal damage to soft and hard dental tissues. This study demonstrates that laser ablation via a 1030-nm Yb:Glass fiber laser can overcome the thermal and mechanical drawbacks observed in the literature, while offering other benefits such as a reduction in clinical time, a reduced ablation area, and a bond strength similar to that of the acid etching without the disadvantages of a wet surface. In addition, the vertical ablation line pattern on the enamel, which is parallel to the direction of tooth brushing, ensures straightforward and effective cleaning after orthodontic treatment.

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