EFFECT OF SURFACE TREATMENT ON ENAMEL SURFACE ROUGHNESS

Değişik Yüzey Tedavi Metotlarının Mine Yüzey Sertliği Bakımından Karşılaştırmalı Olarak İncelenmesi

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ABSTRACT

Purpose: To compare the effects of different methods of surface treatment on enamel roughness.

Materials and Methods: Ninety human maxillary first premolars were randomly divided into three groups (n=30) according to type of enamel surface treatment: I, acid etching; II, Er:YAG laser; III, Nd:YAG laser. The surface roughness of enamel was measured with a noncontact optical profilometer. For each enamel sample, two readings were taken across the sample—before enamel surface treatment (T1) and after enamel surface treatment (T2). The roughness parameter analyzed was the average roughness (Ra). Statistical analysis was performed using a Paired sample t test and the post-hoc Mann-Whitney u test, with the significance level set at 0.05.

Results: The highest Ra (average roughness) values were observed for Group II, with a significant difference with Groups I and III (P<0.001). Ra values for the acid etching group (Group I) were significantly lower than other groups (P<0.001).

Conclusion: Surface treatment of enamel with Er:YAG laser and Nd:YAG laser results in significantly higher Ra than acid-etching. Both Er:YAG laser or Nd:YAG laser can be recommended as viable treatment alternatives to acid etching.

Keywords: Enamel roughness; optical profilometry; Er-YAG laser; Nd:YAG laser; phosphoric acid

ÖZ

Amaç: Farklı yüzey tedavi metotlarının mine sertliği üzerine etkilerinin karşılaştırılması.

Gereç ve Yöntem: Doksan adet insan maksiller birinci premoları mine yüzey tedavi tipine göre rastgele üç gruba ayrıldı (n=30): I, asitle pürüzlendirme; II, Er:YAG lazer; III, Nd:YAG lazer. Mine yüzey sertliği temasta olmayan bir optik profilometre ile ölçüldü. Her bir örnek için yüzey boyunca iki ölçüm alındı—mine yüzey tedavisinden önce (T1) ve mine yüzey tedavisinden sonra (T2). İncelenen sertlik parametresi ortalamala sertlik (Ra). İstatistiksel analiz, anlamli seviyesi 0.05’de, Paired sample t testi ve post-hoc Mann-Whitney U testi kullanılarak yapıldı.

Bulgular: En yüksek Ra (ortalama sertlik) değerleri 1. grup ve III. grupdan anlamlı farklılıklarla 1. grup için gözlemlendi (P<0.001). Asitle pürüzlendirme grubu için Ra değerleri (1. grup) diğer gruplardan anlamli derecede düştü (P<0.001).

Sonuç: Er:YAG lazer ve Nd:YAG lazer ile minenin yüzey tedavisi asitle pürüzlendirmeden anlamli derecede daha yüksek Ra ile sonuçlanmıştır. Hem Er:YAG lazer hem de Nd:YAG asitle pürüzlendirmeye alternative tedavi seçenekleri olarak önerilebilir.

Anahtar kelimeler: Mine sertliği; optik profilometre; Er-YAG lazer; Nd:YAG lazer; fosforik asit
Introduction

A variety of different surface treatment methods are available for orthodontic use. Orthophosphoric acid etching has been widely used to prepare tooth enamel for bonding resins and orthodontic attachments since it was first introduced by Buonocore (1) in 1955. Recently, erbium:yttrium-aluminum-garnet (Er:YAG) and neodymium:yttrium-aluminum-garnet (Nd:YAG) lasers have been proposed as alternatives to phosphoric acid etching. Laser etching of enamel surface leads to a fractured, uneven surface that is ideal for adhesion (2).

Roughness is defined as set of irregularities, ie, small saliencies and re-entries that characterize a surface and can be evaluated by means of electronic appliances, such as a roughness meter. Several studies in the literature have evaluated the roughness of human primary and permanent teeth (3), the effect of microabrasion on the surface roughness of restorative materials, dentin and enamel (4, 5), the correlation between enamel roughness and wettability (6), and the influence of various methods of ceramic surface etching on roughness and the bond strength of metal brackets (7, 8).

However, little or no attention has been paid to the characteristics of the conditioned enamel surface. Enamel conditioning is a fundamental step to achieve good bracket adherence (9). Therefore, the morphologic evaluation of conditioned enamel is important for the analysis and improvement of adhesive systems. Although some studies have investigated the effect of sandblasting and acid-etching on enamel surface roughness (10), only one study has evaluated surface roughness after Er:YAG laser etching (2).

Furthermore, a literature review uncovered no studies which compared the effect of different surface treatment methods (acid etching, Er:YAG laser etching, and Nd:YAG laser etching) on the surface roughness. Given the scarcity of roughness data related to surface treatment methods, the present study aimed to comparatively evaluate the difference in enamel roughness changes after enamel conditioning using 37% orthophosphoric acid etching, Er:YAG laser and Nd:YAG laser etching by means of a non-contact optical profilometer. The null hypothesis was that there would be no difference in surface roughness after the 3 conditioning techniques.

Materials and Methods

Specimen Preparation

The sample consisted of 90 human maxillary first premolars that had been extracted previously. The selected teeth had intact enamel and no caries, cracks, restorations, or hypoplasia. Obtained teeth were stored in 0.1% thymol solution at 4°C until the experiment. The teeth were cleaned from soft tissue remnants and the roots were cut off approximately 2 mm below the cemento-enamel junction. The crowns were then mounted in self-curing acrylic resin, in such a way that the buccal surface of the tooth would be oriented horizontally and about 1-2 mm above the rim of the mould. The mounted teeth were randomly divided into three experimental groups of 30 and a number was assigned to each specimen. The blocks were kept in distilled water at room temperature during the time of the experiment in order to prevent dehydration. A 4x6 mm window was cut in an acrylic resin plate that was used to limit the area of enamel to be treated to the exact dimensions of the orthodontic brackets. One operator held the acrylic plate over the tooth surface while a second operator applied the surface conditioning to the area within the window. Specimens were randomly divided into three groups (n=30) for enamel treatment, as follows:

Group I (acid etching): Teeth were etched for 15 s with a 37% orthophosphoric acid gel (ORMCO, Orange, CA, USA), rinsed for 15 seconds, and dried to a chalky-white appearance for 15 seconds.

Group II (Er-YAG laser irradiation): An Er:YAG laser device (2940-nm wavelength; LightWalker, Fotona, Ljubljana, Slovenia) with an output of 1.5 W was used in medium-short pulse mode (MSP; 100 ms, 120 mj, 10 Hz, 1.5 W). The device uses a fiber-optic system to deliver laser energy to a sapphire tip that is bathed in an adjustable air/water spray. The laser beam was directed perpendicular to the enamel at a distance of 1 mm from the tooth surface and applied for 15 s, with air and water levels set at 90% and 80%, respectively.

Group III (Nd:YAG laser irradiation): An Nd:YAG laser device (1094-nm wavelength; LightWalker, Fotona, Ljubljana, Slovenia) with an output of 1.5 W was used in medium-short pulse mode (MSP; 100 ms, 120 mj, 10 Hz, 1.5 W). The device uses a fiber-optic system to deliver laser energy to a sapphire tip that is bathed in an adjustable air/water spray. The laser beam was directed perpendicular to the enamel...
at a distance of 1 mm from the tooth surface and applied for 15 s, with air and water levels set at 90% and 80%, respectively. After laser ablation, to clear tooth particles and dust, the surface of laser-treated specimens in both group II and III was cleaned with running water without brushing and dried in air.

The surface profile was analyzed at the center of the delimited area using a noncontact optical profilometry (Contour Elite, Bruker Nano Surfaces Division, Tucson, AZ, USA). For each enamel sample, two readings were taken across the sample—before enamel conditioning (T1) and after enamel conditioning (T2). Although perfect repositioning accuracy is impossible at the micron level, the sample was roughly in the same position for every measurement. The roughness parameter analyzed was the average roughness (Ra), which is the arithmetic mean of the height of peaks and depth of valleys from a mean line in the measuring length (Figure 1-4).

![Figure 1. Profilometric image of an intact enamel surface.](image1)

![Figure 2. Profilometric image of an acid-etched enamel surface.](image2)

**Statistical Analysis**

Descriptive statistics including mean and standard deviation were calculated for each group using a statistical software package (MedCalc Statistical Software version 13.0 - MedCalc Software bvba, Ostend, Belgium; http://www.medcalc.org; 2014). All data were examined for normality using Shapiro–
Wilks test. All data was analyzed using a Paired sample t test with the exception of non-normal distribution at T2 in Group I, which was analyzed using Wilcoxon signed rank test. Statistical significance was set at the P<0.05 level.

**Results**

Mean Ra values are summarized in Table 1. No significant differences were found among the mean Ra values of any of the groups at T1 (ANOVA, \( P = 0.970 \)). When compared to the values at T1, Ra values at T2 were significantly higher for all experimental groups (\( P<0.001 \)). Significant differences were observed among mean Ra values of the experimental groups at T2. Mann-Whitney U test showed Group II to have significantly higher Ra values than Group I and III and also Group III to have significantly higher Ra values than Group I (\( P<0.001 \)). The 3D profilometric images of the Er:YAG laser- and Nd:YAG laser-treated enamel surfaces (Figure 3, 4) showed rougher surfaces than those of acid etching (Figure 2).

| Study groups | T1 (Ra) | T2 (Ra) | \( p \) |
|--------------|---------|---------|---------|
| **Group I n=30** | 12.2±0.6 | 15.3±0.5 | <0.001<sup>a</sup> |
| **Group II n=30** | 12.2±0.6 | 19.5±0.4 | <0.001<sup>b</sup> |
| **Group III n=30** | 12.2±0.5 | 17.8±0.5 | <0.001<sup>c</sup> |
| **P = 0.970<sup>d</sup>** | 0.970<sup>d</sup> | <0.001<sup>e</sup> |

<sup>a</sup> Wilcoxon Signed Rank Test, <sup>b</sup> Paired sample t test, <sup>c</sup> Kruskal Wallis test, <sup>d</sup> ANOVA.
Discussion

The current study is the first to compare the effect of three popular orthodontic surface conditioning techniques on enamel roughness by using a non-contact optical profilometer. Our results showed significant differences in the surface roughness data among the groups tested. Therefore, the null hypothesis that there are no differences in surface roughness among the groups must be rejected. All tested surface treatment methods demonstrated significantly greater values in surface roughness parameters than in intact enamel surfaces. This implies increase in surface areas available for bonding. The results of this study also showed both Er:YAG and Nd:YAG laser etching produced rougher surfaces than those of acid etching (Figure 3, 4).

Acid etching is a conventional technique that produces micromechanically retentive structure on enamel by the preferential dissolution of inorganic structure, and then facilitates the penetration of monomers to create resin tags in enamel (1). In the present study, the acid-etch group had the smallest Ra values (Figure 2). This can be explained by the fact that acid etching of uncut enamel surfaces, as for orthodontic bonding, creates a complex profile, caused by the prismless enamel (11). These regions, with parallel and highly packed apatite crystals, create an acid-resistant substrate (12). However, when acid etching cuts enamel surfaces, the carbonate-rich core of the enamel prisms composed of vertically oriented apatite crystals is preferentially dissolved, leaving protrusions at the prism boundaries (typical type I etching) as has been documented in historic scanning electron microscope studies (11, 13). The low Ra values of specimens treated by acid etching may also be attributed to the acid etching’s less effect on the organic materials than those of laser etching (14). The increased roughness observed in both laser groups may be related to the ablation mechanism of the lasers which produce structural modification due to the phase transformation or melting of inorganic substances, expansion of the organic matrix, as well as the subsequent blocking of ion diffusion pathways (15). Nd:YAG laser irradiated samples evaluated by scanning electron microscopy show melted and resolidified surfaces (16, 17). Compared with the Nd:YAG laser, the Er:YAG laser produced rougher surface (Figure 3, 4). This can be explained by an explosive vaporization of water within the tooth during the Er:YAG laser ablation. Er:YAG emits a laser at 2.94 µm wavelength which coincides with a very strong water absorption peak (wavelength 3.0 µm), and which is well absorbed by the OH⁻ group in hydroxyapatite (wavelength 2.8 µm) (18). Light of this wavelength is also absorbed well by enamel (18). When this laser is irradiated on the tooth, the absorbed laser energy is converted to heat and boils water in the tooth. This forms high-pressure steam, and the explosive vaporization of water changes the smooth tooth surface into generally flaky with irregularly serrated and microfissured structure, and one which is usually free from melting and carbonization (14, 19). In addition to producing rougher enamel surfaces, laser systems have advantages of saving chair time and producing acid-resistant surface. The required time for acid etching varies from 15 to 60 seconds. Fifteen seconds of water spraying and 15 seconds of air drying are necessary in phosphoric-acid etching. A total of 45 seconds for each tooth is needed with phosphoric acid. Laser systems are 30 seconds faster than phosphoric-acid etching (only 15 seconds). Thirty seconds of chair-time saving for each tooth equates to at least 5 minutes of chair-time saving for a full-mouth bonding. From a clinical standpoint, saving chair time may also attribute to the improvement of adhesion because it reduces the risk of salivary contamination (11). The surface produced by laser irradiation is also acid resistant. The microfissures and microspaces in the laser-ablated region are believed to trap the free ions essential for remineralization and thereby effectively induces acid resistance in enamel, probably by crystalline improvement and the blocking effect of the organic matrix (14).

The effects of laser etching on surface roughness have been examined in a few previous studies. Sagır et al. (2) found that enamel conditioning with Er:YAG laser produced higher enamel roughness levels than that of the acid etched group by using a contact optical profilimeter. Marquez et al. (20) found an increase in microhardness after Nd:YAG laser irradiation. Similarly, Hess (21) examined the extent of morphologic changes produced in the enamel surface following Nd:YAG laser irradiation by using a scanning electron microscope and showed that the surface had melted and reformed with numerous small, bubble-like inclusions. He suggested Nd:YAG laser as a simple, effective and controlled method of etching the enamel surface of a tooth by altering its surface characteristics (16, 21). Cernavin (22) also reported that Nd:YAG laser caused heat-induced cracking and crazing in irradiated enamel surfaces.
These findings support those of the present study. In contrast, Ariyaratnam et al. (23) evaluated enamel morphology of molar teeth after Nd:YAG laser etching and acid etching by a contact profilometer. They found that no significant difference in surface roughness between laser-treated enamel and acid-etched specimens (23). Although the laser roughened the surface of the enamel (with the formation of microcracks and fissures), it did not provide a surface as retentive as a surface treated with conventional acid etching (23). Arcoria et al. (24) utilized a contact profilometer and showed a moderate amount of roughness approaching that of the acid-etched samples following Nd:YAG laser application, and Kuramoto et al. (25), utilizing a Vickers microhardness test, reported decrease of enamel microhardness with higher delivered energy settings (from 30 to 100 Joules) and non-significant statistical differences of enamel surface microhardness with lower delivered energy (ranging from 3 to 21 Joules). It is difficult to correlate past findings with those of the present study, as methodological differences exist. Also, it should be noted that while studies demonstrating conflicting results in surface roughness of samples subjected to laser irradiation, all the previous studies in the literature utilized a contact profilometer, a Knoop or Vickers hardness test, or scanning electron microscope and all of them examined surface roughness following Er:YAG irradiation or Nd:YAG irradiation, whereas the present study is the first to utilize a noncontact optical profilometer and to compare the roughness following acid-etching, Er:YAG etching and Nd:YAG etching.

Because the roughness value depends on the measurement technique, the investigation protocol used to study surface roughness is important. Surface roughness measurements are performed using Vickers diamond testing machine, contact (or stylus) profilometer, non-contact optical profilometer, or scanning electron microscopes (SEM). The conventional contact profilometers is a linear measurement tool that has often been used to measure roughness, but it produces lower Ra values than does the optical profilometer because of the limitations of the spatial dimensions of its tip in detecting microcracks (26). Moreover, the conventional profilometer may affect the reading or even damage hard dental tissues because of its contact with the specimen (27). Non-contact profilometers generally use some type of laser to scan the surface to create the profile and offer quick measurement of surface features without surface contact. In addition, non-contact profilometers usually generate a surface plane (three-dimensional surface mapping) rather than just simple line profiles, which allows volumetric loss analysis (28). In comparison to contact profilometry, the optical method does not risk damage to the sample surface, which could provide higher reliability for repeated measurements (29). SEM assesses enamel roughness qualitatively with visual analysis and therefore the evaluation of roughness of enamel surfaces from SEM photomicrographs can be unreliable and subjective (30). Non-contact profilometry has the advantage of measuring the absolute depth of the defects over the electron microscopy (31). To measure the surface roughness in the present study, a non-contact optical profilometer was chosen because this device gives repeatable, quantitative metrology data and also 3D color image of the specimens to reveal microscopic details.

Conclusion

The results of this study suggest that Nd:YAG laser and Er:YAG laser operated according to the parameters used here significantly increases buccal enamel roughness and produces more roughness than does conventional phosphoric acid etching. Therefore, both Nd:YAG laser and Er:YAG laser may be a reasonable alternative to phosphoric acid treatment especially for treating teeth that are exceptionally vulnerable to acid.

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Conflict of interest
None declared

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