Selcuk Dental Journal 11/3 (Aralık 2024): 309-312

Research / Araştırma Makalesi

Chemical Analysis of Femtosecond Laser Irradiated Enamel

Femtosaniye Lazer ile Pürüzlendirilen Minenin Kimyasal Analizi

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ABSTRACT

Aim: The aim of the study was to assess the chemical alterations in enamel after femtosecond laser irradiation.

Material and Methods: Fifteen permanent molars were used in the experiment, with 3x3 mm² areas prepared on the buccal surfaces of each tooth. These areas were irradiated using a Ti laser system (800 nm, 90 fs, 1-3 kHz) for 15 seconds. To assess the chemical composition of the samples before and after laser treatment, energy dispersive X-ray spectroscopy (EDX) was used. This analysis measured the mean weight percentage (wt%) of minerals including calcium (Ca), phosphorus (P), carbon (C), aluminum (Al), oxygen (O), magnesium (Mg), potassium (K), and sodium (Na).

Results: The mean wt% of all tested minerals were changed. While the change in O, P, Al ions was found to be significant ($p < 0.05$), the change in Ca, Mg, Na, C was found to be insignificant ($p > 0.05$).

Conclusion: Our results indicate that femtosecond laser irradiation influences the mineral composition of the enamel.

Keywords: Femtosecond laser, Mineral content, Enamel

ÖZ

Amaç: Bu çalışmanın amacı femtosaniye lazer ile ışınlanmış minenin kimyasal değişimlerini değerlendirmektir.

Gereç ve Yöntemler: On beş molar diş kullanıldı. Dişlerin bukkal yüzeylerinde 3x3 mm2'lik alanlar hazırlandı. Hazırlanan yüzeyler Ti: Sapphire lazer sistemi (800 nm, 90 fs, 1-3 kHz) ve 15 s süre ile ışınlandı. Örneklerin işlem öncesi ve sonrası kimyasal içerik analizleri enerji dağılımlı X-ışını spektroskopisi (EDX) ile yapıldı. Kalsiyum (Ca), fosfor (P), karbon (C), alüminyum (Al), oksijen (O), magnezyum (Mg), potasyum (K), sodyum (Na) mineral içeriğindeki ortalama ağırlık yüzdesi (ağırlıkça %) değerlendirildi.

Bulgular: Test edilen tüm minerallerin ağırlıkça ortalama % değerleri değişmiştir. O, P, Al iyonlarındaki değişim önemli bulunurken ($p < 0.05$), Ca, Mg, Na, C'deki değişimler eöenmli değildir ($p > 0.05$).

Sonuç: Bulgularımız minenin mineral içeriğinin femtosaniye lazer ışınlamasından etkilendiğini göstermektedir.

Anahtar Kelimeler: Femtosaniye lazer, Mineral içeriği, Mine

Introduction

With the introduction of adhesive dentistry, the importance of the enamel bonding basis has been understood. It turned out to strongly influence the development of dental adhesive systems.[1] Roughening the enamel layer is the most reliable approach for achieving durable enamel bonding.[2] The technique of roughening the enamel layer with acid etching was introduced by Buonocore.[3] Today, this classical technique for etching the enamel layer is widely used in composite fillings, pit-fissure sealant applications and bracket bonding in orthodontics.[4] As a result of etching with acid, selective dissolution occurs in the enamel as micropores. This results in enamel loss from 10 µm to 50 µm deep.[5] The primary drawbacks of acid etching include the risk of decalcification, which can leave tooth enamel vulnerable to cavities, the roughened surface being easily contaminated, improper washing and drying negatively affecting the bond strength.[6] In addition, enamel discoloration and bad taste are other negative aspects of this method. New searches were undertaken to eliminate these disadvantages.[7]

Laser irradiation was used to roughen the enamel and dentin layers. Various laser types such as (Er: YAG, Er,Cr:YSGG, Nd:YAG, CO_2) have been used for this purpose until today.[8,9] The impact of these laser treatments on the enamel layer included melting and recrystallization. The advantages of laser etching over acid etching are that it reduces the risk of demineralization and can be applied in a shorter time. In addition, the tooth does not need to be isolated from moisture.[9,10] However, previous studies have proven that during application these lasers produce undesirable thermal side effects, cracks and consequent lack of bonding.[8,10,11] Ti: Sapphire laser system, which has been studied in dental applications in recent years, has surpassed other lasers with its features such as sensitive tissue removal and minimum thermal damage.[12,13] A femtosecond laser can deliver ultra short laser pulse that results in cold ablation within a thermal relaxation time. In addition, the femtosecond laser protects the tissue with crack-free and residue-free cold ablation, has the advantages of good surface morphology and no reshaped layer formation.[14,15] Ablation of enamel with femtosecond lasers has been studied by several authors[16-18] but few studies have performed chemical composition analysis.[19,20] In this study, the chemical changes caused by femtosecond laser etching in the enamel layer were investigated. The null hypothesis is that there is no change in mineral content before and after roughening.

Material and Methods

Experimental Design

The study sample comprised fifteen enamel specimens, which underwent femtosecond laser irradiation. The quantitative outcome measured was the chemical alteration of enamel, evaluated through energy dispersive X-ray spectroscopy (EDX) analysis.

Preparation of specimens

Teeth with caries lesion or restoration, enamel crack defects, and fluorosis were not included in the study. Organic deposits on all viable molars were eliminated using a periodontal scaler. After being cleaned and rinsed with fluoride-free pumice, the teeth were kept in distilled water at room temperature until the experiment commenced. The roots were embedded 2 mm apical to the cemento-enamel junction. After manually polishing the teeth's enamel surfaces with 600- and 1200-grit silicon carbide paper discs, the samples were air dried. The buccal surfaces were coated with nail varnish except for a 3 × 3 mm² patch that remained exposed in the center third.

Femtosecond laser application

The Ti: sapphire laser system included a pumped oscillator (Quantronix, Ti: Light, NY, USA) followed by an ultra-fast amplifier (Integra-C-3.5, NY, USA). This system operates at a wavelength of 800 nm with a pulse duration of approximately 90 fs and a repetition rate of 85 MHz. The

Gönderilme Tarihi/Received: 15 Eylül, 2024
Kabul Tarihi/Accepted: 23 Ekim, 2024
Yayınlanma Tarihi/Published: 23 Aralık, 2024
Atıf Bilgisi/Cite this article as: Özüdoğru S, Kahvecioğlu F, Tosun G. Chemical Analysis of Femtosecond Laser Irradiated Enamel. Selcuk Dent J 2024;11(3): 309-312 Doi: 10.15311/ selcukdentj.1550482

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Doi: 10.15311/ selcukdentj.1550482

pulse power applied was 500 mW.[28] A micromachining system (Quantronix, QMark, USA) was used for scanning, with the laser beam positioned at the focal point. The calculated laser intensity for each sample in the study was 9.82×10^{14} W/cm^2. Additionally, the micro-processing system employed a galvo scanner with an f-theta lens design, a scanning speed of 1 mm/sec, and a jump speed of 125 mm/sec. The pump laser system had a pulse repetition rate of 1–3 kHz at 800 nm.

Chemical analysis test

EDX was utilized to measure the weight percentage of elements such as calcium (Ca), phosphorus (P), carbon (C), aluminum (Al), oxygen (O), magnesium (Mg), potassium (K), and sodium (Na) in an enamel area of 300 × 300 µm in all samples, both prior to and following femtosecond laser irradiation (Zeiss EVO LS 10; Carl Zeiss SMT Ltd., Cambridge, United Kingdom).

Statistical analyses

SPSS 22.0 for Windows was utilized to conduct the statistical analysis (SPSS, Chicago, IL). The paired sample t test by Wilcoxson test was used to examine the mineral content of enamel both before and after laser irradiation. A significance threshold of $p<0.05$ was established.

Results

In femtosecond laser irradiation, the change in Ca, Mg, Na, C ions according to the average weight percent was not found significant ($p>0.05$). A significant increase in O rate was detected ($p=0.009$). The decrease in P and Al ratios was significant ($p=0.028$, $p=0.029$) (**Table 1**).

Table 1. Mean Percentage Weights of the Elemens

	Femtosecond Laser Irradiation				
	Before Application		After Application		p
	Mean.±S.S.	Min.-Max.	Mean.±S.S.	Min.-Max.	
Ca	39,79±1,02	33,59-44,52	37,20±1,46	26,71-43,56	0,103
O	41,20±1,71	33,86-49,49	49,24±1,44	40,81-54,74	**0,009**
P	14,70±1,51	6,73-20,00	9,69±1,42	4,51-16,46	**0,028**
C	3,26±0,70	0,79-8,09	3,12±2,19	0,22-22,85	0,952
K	0,19±0,02	0,12-0,34	0,20±0,03	0,02-0,40	0,766
Mg	0,09±0,03	0,00-0,35	0,02±0,01	0,00-0,13	0,125
Al	0,10±0,03	0,00-0,34	0,01±0,006	0,00-0,05	**0,029**
Na	0,39±0,09	0,00-0,98	0,47±0,04	0,29-0,65	0,472

P<0.05
Ca, calcium; O, oxygen; P, phosphorus; C, carbon; K, potassium; Mg, magnesium; Al, aluminum; Na, sodium.

Discussion

The mineral composition of teeth is primarily made up of hydroxyapatite (chemical formula $Ca_{10}(PO_4)_6(OH)_2$), along with water and organic substances like proteins and lipids. Approximately 96% of enamel's mineral content is composed of inorganic compounds by weight, while the remaining 4% consists of organic compounds and water.[21]

Our study examined the femtosecond laser system for roughening of tooth enamel in terms of chemical elemental analysis. In the study, significant changes were found in mineral contents and the null hypothesis was rejected.

Irradiation with a femtosecond laser, which generates femtosecond optical pulses (1 fs = 10 − 15 s), is one new surface treatment technology.[28] Less microcrack formation and the removal of delicate dental hard tissues using ultrashort laser pulses that cause little to no heat damage are the most efficient advantages offered by femtosecond lasers.[14] Research on femtosecond laser systems has looked into a number of parameters for dental applications, such as wavelength, repetition rate, alternative laser medium, and pulse duration.[14,25,28] There were a limited number of studies investigating the chemical changes in the enamel layer caused by femtosecond laser ablation.[19,22] Both very low (1 kHz) and very high (500 kHz) laser frequencies were used in these investigations. The ablated dentin or enamel did not sustain any collateral damage when a 50 kHz laser frequency was utilized.[25] Another study showed that enamel could be successfully ablated at a laser frequency of around 100 fs (λ ~ 800 nm) at 1 kHz without showing signs of heat effects or cracking.[23] In the present study 1–3 kHz; 90fs; 800 nm parameters were used.

Studies that conducted chemical analysis of the enamel surface exposed to various types of lasers reported a reduction in carbon content.[22,23] In our study, a decrease in C ratio was observed in the applied power value. Although carbonate is found in the structure of hydroxyapatite, it is the underlying cause of the first enamel caries lesions. When considering chemistry, carbonate is believed to have a destabilizing effect. A low carbonate content in enamel is a key indicator that helps explain the preservation of the subsurface region in areas affected by caries. This is a great advantage for the laser roughened enamel surface. It is an indication that a layer resistant to caries has formed.[24]

The carbon and phosphorus contents did not change significantly ($p>0.05$) following laser treatment, but there was a corresponding rise in oxygen and a decrease in carbon, in line with our study by Petrov et al.[25] The cleaning action of laser ablation, which eliminated surface contaminants, was credited with the decrease in carbon concentration. This might actually be a sign that using a laser instead of another technique can improve adhesion to the enamel surface.

Casarin et al.[26] concluded that following 15 seconds of femtosecond laser irradiation at 1 W power on the enamel surface, there was a notable increase in the area value of phosphate peaks, along with a reduction in both carbonate content and the carbonate/phosphate ratio, while the enamel morphology remained unchanged. However, no significant change was found in the phosphate ratio in our study. This is thought to be due to the differences in the parameters used. The parameter used in the present study is a smaller power.

On the contrary, in a study by Quang-Tri Le et al.[27], in which they examined the surface topographic, compositional, and structural changes after femtosecond laser ablation of the enamel at power up to 14 j/cm^2, a layer of resolidified material was seen to cover the ablation surfaces, indicating that hydroxyapatite melts in conjunction with ablation. This layer showed pores and burst gas bubbles that were caused by the liquid phase's emission of CO2 and H2O, which are the gaseous breakdown products of hydroxyapatite. Furthermore, amorphous calcium phosphate was found in the resolidified sample by Micro-Raman examination. Unlike this study, the free Ca ratio did not increase in the current study. In addition, a decrease in the amount of C was observed. This shows that the laser parameter used in this study does not cause melting of hydroxyapatite and there is no thermal effect.

The authors of this article are aware of the limitations (one laser operating parameter of the experiment, no thermal cycling). There are few studies in the literature on this subject and more studies are needed to introduce routine clinical use by examining different laser parameters.

Conclusion

Femtosecond laser irradiation has the potential to induce chemical changes in tooth enamel. Additional research is needed to further understand the laser-induced alterations in enamel composition.

ACKNOWLEDGEMENTS

The authors thank to Prof. Dr. Hamdi Sükür Kılıç and Assist. Prof. Yasemin Gündoğdu both laser physicists, for their valuable insights and contributions.

Değerlendirme / Peer-Review

İki Dış Hakem / Çift Taraflı Körleme

Etik Beyan / Ethical statement

Bu çalışmanın hazırlanma sürecinde bilimsel ve etik ilkelere uyulduğu ve yararlanılan tüm çalışmaların kaynakçada belirtildiği beyan olunur.

It is declared that during the preparation process of this study, scientific and ethical principles were followed and all the studies benefited are stated in the bibliography.

Benzerlik Taraması / Similarity scan

Yapıldı – ithenticate

Etik Bildirim / Ethical statement

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Finansman / Grant Support

Yazarlar bu çalışma için finansal destek almadığını beyan etmiştir. | The authors declared that this study has received no financial support.

Çıkar Çatışması / Conflict of Interest

Yazarlar çıkar çatışması bildirmemiştir. | The authors have no conflict of interest to declare.

Yazar Katkıları / Author Contributions

Çalışmanın Tasarlanması | Design of Study: SÖ (%30), FK (%30), GT (%40)
Veri Toplanması | Data Acquisition: SÖ (%30), FK (%30), GT (%40)
Veri Analizi | Data Analysis: SÖ (%30), FK (%40), GT (%30)
Makalenin Yazımı | Writing up: SÖ (%40), FK (%30), GT (%30)
Makale Gönderimi ve Revizyonu | Submission and Revision: SÖ (%40), FK (%30), GT (%30)

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