

# Evaluation of dentin topography after Er, Cr: YSGG laser irradiation and phosphoric acid etching using AFM, SEM and contact angle

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#### Abstract

**Objective:** The goal of this in vitro investigation is to assess laser irradiation and phosphoric acid etching effects on the dentinal surface topography.

**Materials and Methods:** A total of 48 sound human premolars extracted for orthodontic purposes were included in this study. Teeth were divided into four groups (n = 10), G1 without surface treatment, G2 etched with phosphoric acid 37% for 15 seconds, and G3 and G4 were treated with an Er, Cr:YSGG laser at the following settings: 1W, 1.5W, respectively with set other laser parameters at 20 HZ and 10% air/water ratio. The effects were evaluated using SEM, AFM, and contact angle.

**Results:** AFM and SEM images showed changes in the surface topography of the dentin with an increase in the surface roughness measurements and devoid from smear layer in the laser-treated groups compared to the control and phosphoric acid-treated groups. Also, highly significant differences ( $p \le 0.01$ ) were shown in the laser irradiation groups, specifically G3 with 1W, when compared with others considering contact angle measurement.

**Conclusion:** Er, Cr: YSGG laser can be used as an alternative to conventional phosphoric acid etching for etching and preparing dentin surfaces prior to restoration or bracket placement.

Keywords: Er, Cr: YSGG, phosphoric acid, dentin, SEM, and wettability test.

### 1. Introduction

Dentin adhesion is thought to be a more complex and difficult process than enamel adhesion because of its chemical composition, humidity, and morphological diversity. Furthermore, pathological and physiological processes might create morphological and biomechanical changes in the dentin is also unique [1]. In the conventional method, the smear layer is removed from the dentin surface by acid etching which also leads



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to exposing collagen fibrils and dentinal tubules; this could help in resin monomer infiltration. Subsequently, hybrid layers form during the polymerization of the infiltrating resin monomers [2].

Acid etching of dentin has some shortages, such as increased dentin permeability and wetness, with an increased possibility of pulpal irritation and collagen denaturation, in addition to the time and risks or precautions [3]. In recent years, there has been a great interest in the use of lasers in dentistry for different purposes and applications, such as cavity preparation, caries prevention and detection, teeth bleaching, and dental implants [4, 5]. Because of its strong absorption by water and hydroxyapatite crystals, the Er, Cr:YSGG laser may effectively ablate enamel and dentin [6]. By providing uneven surfaces and open dentinal tubules without a smear layer, Er, Cr:YSGG laser irradiation with modified laser parameters or technique may generate energy suitable dentin surface adhesion [7]. When Er, Cr:YSGG laser energy is absorbed by tooth layers components, it causes evaporation of water, an increase in internal pressure, and microexplosion, resulting in the formation of a rough dentin surface [8-10]. Depending on the type and energy of laser applied to the surface, laser etching produces surface irregularities and roughening to a depth of 10 to 20 micrometers, which is comparable to that produced by the acid [11]. According to different laser applications in the dental field, laser etching might be suggested with advantages over traditional acid etching, including painlessness, the absence of vibration or heat generation, the creation of an uneven and fractured surface suited for adhesion, the absence of a smear layer, and increased tooth resistance to caries attack [12-14]. Er, Cr: YSGG Laser widely used in dentistry for different dental procedures, including cavity preparation [15], disinfection of prepared cavity and root canal [16, 17], and in bonding of esthetic restoration without thermal damage to dental pulp [18, 19].

The goal of this study is to estimate how much Er, Cr: YSGG laser is a suitable alternative source for phosphoric acid etching in dentin.

#### 2. Material and Method

#### 2.1 Sample Collection and Preparation

A total of (48) intact premolars were collected from different health centers and clinics in Baghdad. The volunteers were in the age range (18-35), and teeth were extracted for orthodontic treatment. Primarily, teeth were cleaned from blood, tissue remnants, and debris with dental scalar and prophylaxis dental paste; they were stored in 1% thymol solution until used. Preparations of samples were performed with the diamond disk, which is used to cut the occlusal surface of teeth to a level of central groove and expose the dentin surface.

The cutting procedure was done with the aid of a dental surveyor (Dentaurum para therm, German) and water irrigation to avoid any deterioration to the sample surface from hat generation during cutting; this also helped in the standardization of the procedures. The acrylic block was constructed with a cylindrical silicon mold of about 2.5 cm in length and 2 cm in diameter, as seen in Figure 1. Samples were assigned into four groups:-

G1: Samples without surface treatment

G2: Samples were treated with 37% orthophosphoric acid etchant

G3: Samples were treated with Er, Cr: YSGG laser (1W, 20 HZ, 10% air, and water ratio, 60µs (pulse

duration), 66sec (irradiation time), 20mm<sup>2</sup> (irradiation area)

G4: Samples were treated with Er, Cr: YSGG laser (1.5 W, 20 HZ, 10% air, and water ratio, 60µs (pulse

duration), 66sec (irradiation time), 20mm (irradiation area)

Laser parameters in G3 and G4 were selected according to the result of the pilot study.





**Fig.1:** Sample Preparation Procedure (a) de-cusped occlusal surface with diamond disk and dental surveyor (c) acrylic block construction.

# 2.2. Etching Application

#### A. Phosphoric Acid Application

Dentin samples of G2 were etched with 37% orthophosphoric acid gel (3M, ESPS, USA) for 15 seconds and rinsed vigorously with water for 20 seconds according to the manufacturer's instructions.

#### B. Laser Application

Er,Cr:YSGG laser with the MZ6 laser tip (WaterLase iPlus, USA) was used to irradiate specimens of G3 and G4. At a stand-off distance of about 2mm between the laser tip and the sample surface, the laser beam was directed perpendicularly to the dentin surface. For standardization of laser applications, laser irradiation was performed with the aid of a CNC machine (computerized numerical control unit) with a scanning speed of 0.3 mm/sec, as seen in Figure 2.



Fig.2: Laser irradiation of specimen surface with the aid of CNC machine.



#### 2.3. Light Microscopic Evaluation

All samples were examined with a light microscope (Euromex microscope, Netherlands) before and after acid agent application and laser irradiation to evaluate their effects on the dentin surface.

#### 2.4. SEM Examination

One sample representing each group was examined with SEM (Inspect F-50, FEI Electron Optics International B.V., Netherlands). Before being inspected with an SEM machine, the samples were sputtered with gold in a vacuum chamber. The morphological examination of the dentin surface was done.

#### 2.5. AFM Evaluation

For more precise information about the surface changes and roughness of dentin, one sample from each group was examined with AFM (Nano surf, Liestal, Switzerland); the root mean square of surface roughness is displayed in contact mode by the atomic force microscope's scanning probe.

#### 2.6. Assessment of Contact Angle

Ten samples from each group were examined with a contact angle instrument (CAM 110, Taiwan). The instrument was connected to a computerized digital camera to capture an image of the water drop on the sample surface within (30) seconds. The water drop was about  $2\mu$ l, and the measurement was done at room temperature. Statistical analysis was performed using SPSS, version 24, at p≤0.05.

#### 3. Result

#### 3.1. Light Microscopic Evaluation

Light microscopic evaluation showed that there are scratching lines on the dentin surface for G1, which occurred during sample preparation, while for G2, the dentin surface was presented with a superficial effect of acid etching, and for laser groups, the G3 and G4 dentin surface had a uniform pattern of laser effect and surface irregularity as shown in Figure 3.

#### 3.2. SEM Evaluation

SEM images for G1 showed high dental debris due to the sample preparation procedure, as shown in Figure 4a. For G2, the dentin surface was presented with partially occluded dentinal tubules and covered with a smear layer, as seen in Figure (4,b), while laser-irradiated groups (G3 and G4) were presented with clean, highly irregular surfaces devoid of a smear layer, as in Figures (4c, and d).

#### 3.3. AFM Evaluation

Atomic force microscopy (AFM) evaluation showed that laser-irradiated groups (G3 and G4) have the highest value of surface roughness (Ra) compared to other groups (G1 and G2), as seen in Figures 5 and Table 1.

Groups	Ra (nm)
G1	60.82
G2	84.48
G3	404.5
G4	116.3

**Table 1.** Surface roughness using Atomic Force Microscopy for tested groups.





**Fig .3:** Light Microscopic Images of G1 (a) untreated dentin, G2 (b) acidic etched dentin, G3(c) laser irradiated dentin, and G4 (d) laser irradiated dentin at 4X magnification power.



**Fig.4:** SEM Images of G1 (a) untreated dentin, G2 (b) acidic etched dentin, G3(c) laser irradiated dentin and G4 (d) laser irradiated dentin at 1300Xmagnification power



**Fig .5:** AFM Images of G1 (a) untreated dentin, G2 (b) acidic etched dentin, G3(c) laser irradiated dentin, and G4 (d) laser irradiated dentin.

#### 3.4. Contact Angle Evaluation

Descriptive statistical analysis showed that the highest mean value of contact angle was in G1 (72.83) (without surface treatment), followed by G2 (48.18) (acid-treated samples), and the lowest mean of contact angle was in G3 (36.81) and G4 (37.13) Er,Cr:YSGG laser-irradiated groups. Statistical analysis using the ANOVA test showed a highly significant difference between the tested groups at ( $p\leq0.01$ ), as shown in Table 2. Tukey (HSD) test was performed for making a comparison between each two groups regarding contact angle. The result showed a highly significant difference between all groups, with no significant difference between the laser-irradiated group (G3-G4), as shown in Table 3.

Table2. Descriptive statistics and ANOVA test for contact angle in the tested groups.

Groups	Mean± SD	F-test				
		Sum of Squares	D.f	Mean Square	F-value	P- Value
Gl	72.83±2.09	4491.476	3	1122.869	93.400	.000
G2	48.18±1.39	240.442	36	12.022		
G3	36.81±1.61	4731.918	39			
G4	37.13±1.18					



Tukey HDS				
(I)	(J)	(I-J) Mean difference	Sig	
G1	G2	24.65*	.000	
	G3	36.02*	.000	
	G4	35.70*	.000	
G2	G3	11.37*	.001	
	G4	$11.05^{*}$	.000	
G3	G4	1.68	1.000	

**Table3.** Tukey HSD test between each two groups.

#### 5. Discussion

To reestablish function and aesthetics within the principles of optimum tooth structure preservation, bonding to hard dental tissues has been regarded as crucial in operative dentistry. Different dentin surface treatments can have an impact on the efficacy of dentin bonding techniques. [20]. The SEM, AFM, and light microscopic examination results of this in vitro study indicated an increase in surface roughness and irregularity of dentin samples in the laser-treated groups compared with the control and acid-treated groups. The increased surface roughness could be attributed to the different mechanisms of action of laser (thermal) and acid (chemical). The interaction of the Er:Cr:YSGG laser with the dentin's water and hydroxyapatite causes microexplosion and ejection of tooth particles that build on the dentin surface, resulting in a roughened surface [8]. Also, the increase in surface roughness in laser-treated dentine could be attributed to the absence of a smear layer, the presence of exposed dentinal tubules, and chimney-like formations. Furthermore, the laser may selectively remove the intertubular dentin by ablation rather than peritubular dentin since this site contains more water and a lower mineral concentration, which results in projecting dentinal tubules with a cuff-like appearance [21–23]. This research result was in line with Benderli et al., Mahdisiar et al., and Issar et al. [24–26], who found that exposure of dentin surfaces to the Er,Cr,YSGG laser produced a sponge-like appearance and a microscopically rough surface, which might be indicators of the melting and re-crystallization processes. While these findings disagree with those of Al Habdan et al. [27], which could be explained by higher power employed in their experiment (4.5W, 50Hz, and a tipto-specimen distance of 0.5 mm). High power and frequency (4.5W, 30-50Hz) may result in high energy and temperature on the tooth surface, causing melting and resolidification and diminishing surface roughness. This is partially seen when G3 compared to G4 laser groups; higher energy delivered to the dentin surface may generate more heat, which leads to melting of the peritubular dentin. Acid etching destroys intertubular dentin, removing chimney-like structures and expanding the orifices of dentinal tubules. Furthermore, sequential acid etching reveals unknown depths of the demineralization zone [28]. The role of wettability in the stability of bonding between dentin and restoration is crucial. The depth of adhesive infiltration and dentin wettability influence the quality and durability of the resin-dentin hybrid layer [29]. A statistical analysis of the wettability test revealed that laser groups have a lower contact angle than the control and acid-etched groups. This result is related to the fact that when the surface roughness increases, the contact angle decreases, and the wettability is improved [30–32]. Surface irregularity and the existence or extent of the smear layer have a high impact on the surface's wettability [33]. The viscosities of a solution, surface roughness, and heterogeneity all have an effect on the contact angle [34]. The effect of laser in dental applications can be controlled by two groups of variables: the first is laser parameters such as power, wavelength, pulse duration, irradiation mode, and time, which are under the dentist's control, and the second is dentin optical properties, which differ between individuals [35]. Although trained dental personnel can still use the second set of criteria, they benefit from the freedom to choose laser parameters to achieve the purpose or goal of laser use.



#### 6. Conclusions

Based on the findings of this study, Er, Cr: YSGG laser showed improvement in dentin surface topography and can be utilized as an alternate and safe method for surface etching. Laser parameters, specifically the power, are a major contributing factor in surface modification; 1W showed the best result considering surface roughness and wettability.

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# تقييم تضاريس العاج بعد التشعيع بالليزر والحفر بحمض الفوسفوريك عن طريق الفحوص المجهريه الدقيقه وزاوية التلامس

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#### الخلاصة

**الهدف:** تم إجراء هذه الدراسة البحثية لتقييم تأثير إشعاع الليزر والحفر بحمض الفوسفوريك على تضاريس سطح العاج. ا**لطرق:** تم استخدام (48) ضواحك بشرية سليمة تم قلعها لغرض تقويم الأسنان. ثم قسمت إلى أربع مجموعات (العدد = 10) G1 بدون معالجة سطحية، G2 محفور بحمض الفوسفوريك 37% لمدة 15 ثانية، G3 و G4 تمت معالجتها بالليزر Er,Cr:YSGG باستخدام المعلمات التالية (1 واط، 1.5 واط، 20 هرتز ونسبة هواء وماء 10٪).

النتائج: أظهرُت صور الفحوصات المجهرية زيادة في خُشونة سطح العاج بدون طبقة اللطاخة للمجموعات المعالجة بالليزر مقارنة بمجموعتي السيطرة والمعاملة بالفوسفور، كما أظهرت المجموعات المعالجة بالليزر فرق كبير للغاية مقارنة بالمجموعات الأخرى في زاوية التلامس.

ا**لاستُتَاجاتٌ:** يمكن استخدام ليزر الاربيوم كروميوم كبديل للنقش التقليدي بحمض الفوسفوريك لحفر وتحضير سطح الأسنان قبل الحشوات الضوئيه وتقويم الاسنان.

