

MORPHOLOGY AND COMPOSITION ANALYSIS OF ENAMEL SURFACE WITH DENTAL ADHESIVE FOLLOWING THE APPLICATION OF ND:YAG ABLATION

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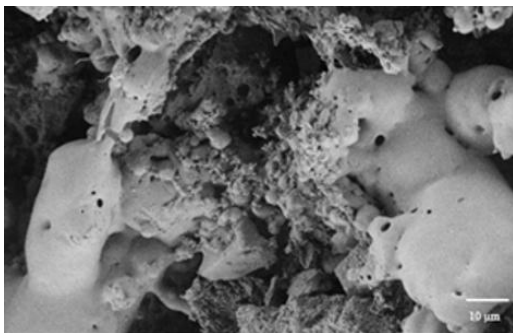
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Graphical abstract



Abstract

Nd:YAG laser with a wavelength of 1064 nm has been used for various applications in dentistry, including for soft tissue and hard tissue applications. This study aimed to investigate the changes in morphological structures and elemental composition of enamel surface after composite removal using energy variations of Nd:YAG laser. 12 healthy human premolar teeth were cut into half, and Blüglöo adhesives were applied to the tooth surface. The samples were subjected to Nd:YAG laser irradiations with three different energy parameters, 510 mJ, 540 mJ, and 580 mJ. The changes in enamel surface morphology and composition of elements were analyzed using Field Emission Scanning Electron Microscopy (FESEM) and Energy Dispersive X-Ray (EDX). Surface morphology indicates that 540 mJ can potentially be used for composite adhesives removal. For the elemental composition, carbon, phosphorus, and calcium were statistically significant between samples without composite, after bracket debonding, and after laser irradiation. Several morphological changes may occur on the enamel surface after samples were irradiated with a laser. Energy parameter of the laser plays a vital role towards the desired surface. In this study, 540 mJ is seen to be potential for material removal process on the enamel surface.

Keywords: Nd:YAG laser, enamel, scanning electron microscopy, laser ablation, dental

Abstrak

Laser Nd:YAG dengan panjang gelombang 1064 nm telah digunakan untuk pelbagai aplikasi dalam pergigian, termasuk aplikasi untuk tisu lembut dan

tisu keras. Kajian ini bertujuan untuk mengkaji perubahan struktur morfologi dan komposisi elemen permukaan enamel selepas penyingkiran komposit menggunakan variasi tenaga laser Nd:YAG. 12 gigi premolar manusia yang sihat dipotong menjadi setengah, dan pelekat Blüglöo telah diletakkan pada permukaan gigi. Sampel dikenakan sinaran laser Nd:YAG dengan tiga parameter tenaga yang berbeza, 510 mJ, 540 mJ, dan 580 mJ. Perubahan morfologi dan komposisi permukaan enamel telah dianalisis dengan menggunakan Mikroskopi Pengimbasan Pelepasan Elektronik (FESEM) dan Energy Dispersive X-Ray (EDX). Morfologi permukaan menunjukkan bahawa 540 mJ berpotensi digunakan untuk penyingkiran pelekat komposit. Untuk komposisi unsur, karbon, fosforus, dan kalsium didapati signifikan secara statistik di antara sampel tanpa komposit, selepas bukaan pendakap, dan selepas penyinaran laser. Beberapa perubahan morfologi berlaku di permukaan enamel selepas sampel disinari dengan laser. Tenaga laser memainkan peranan penting terhadap permukaan yang dikehendaki. Dalam kajian ini, 540 mJ dilihat berpotensi untuk menyingkirkan bahan pelekat pada permukaan enamel.

Kata kunci: Laser Nd:YAG, enamel, pemeriksaan mikroskop elektron, ablas laser, pergigian

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1.0 INTRODUCTION

Lasers have been widely used in dentistry for various applications and dental procedures such as cavity preparation [1], caries removal [2] as well as diseased gingival tissues removal. The advantages of a laser during treatment, such as able to reduce pain, less time of treatment, and less vibration to the patient [3, 4], make it more favourable compared to the conventional approaches.

Four types of laser-tissue interactions that might happen after a laser beam reaches a tissue surface include reflection, refraction, scattering, absorption or transmission [5]. These different interactions probably occurred on the surface, depending on the optical properties of tissue and laser parameters used, such as energy, wavelength, pulse duration, and operation mode [6]. The penetration depth of laser radiation in the target surface also determines a laser's suitability for surgical procedures [7].

Photothermal effects, photochemical reactions, photomechanical interactions, and photoelectrical interactions are the examples of laser interaction mechanisms after biological interface occurs between laser light and target surface [8]. During laser irradiation, water molecules and organic components of biological tissues will selectively absorb the energy from the laser, which causes them to evaporate and produce heat (thermal energy) from the process called photothermal evaporations [9].

Photoablation is a kind of photothermal interaction that remove the material by the vaporization process. When laser light bombarded the target surface, the absorption of thermal energy happened on the surface and led to a heating process [10]. The

heating condition eventually caused melting and vaporization of the target material [11]. The light effect from the photon results in temperature rise, which then converted into kinetic energy and formed vibrational relaxations and non-radiative processes [12].

Orthodontic treatment involves the use of braces that are bonded to the enamel surface with the help of arc wires threaded through the bracket [13]. Dental materials such as resin ionomer cement, composite resin fillings, and dental amalgam fillings function to bond the orthodontic brackets to the tooth surface [14]. During the material removal process, the laser acts as a medium to ablate the residual material from the enamel surface, instead of using mechanical and conventional techniques that can cause discernable damage to the tooth surface [15]. A study by Siniaeva *et al.* [16] indicates positive results of material removal using a microsecond Nd:YAG laser, without causing any severe damage to the tooth surface. Another study by Alexander *et al.* [17] showed that 355 nm laser pulses with a pulse width of 10 nanoseconds able to remove the composite adhesive through selective laser ablation.

However, choosing the correct laser energy parameter is crucial, particularly to prevent any microstructural damage to the enamel. Appropriate selection of laser exposure may efficiently remove materials while minimizing unnecessary exposure to the surrounding tissues. Additionally, composite materials produce various reactions to laser irradiation, such as the presence of bubbles, melting effect, recrystallization, evaporation, pores, and other outcomes [13, 16, 18]. Thus, the objective of this study is to investigate the morphological changes and elemental composition of the enamel surface

following Nd:YAG laser removal of composite adhesives based on the energy variation of the laser.

2.0 METHODOLOGY

2.1 Sample Preparation

Twelve healthy and non-carries premolars were collected in this study. The teeth were extracted due to orthodontics procedure. The extracted teeth were collected at the Advanced Medical and Dental Institute (AMDII) dental clinics and stored in a 70% normal saline solution (B Braun, Germany) at the room temperature. The collection of the samples was approved by the Human Research Ethics Committee of the Universiti Sains Malaysia (USM). A random selection of teeth samples was done after the samples were cleaned from any caries and debris. The teeth were longitudinally sectioned into two parts, and the root parts were cut using a low-speed precision saw (Buehler, USA). After the cutting process was done, the teeth were mounted using Epofix solution (Struers, Denmark) to provide a stable and fixed position for the teeth samples. The bottom part of the mounted teeth was ground using MetaServ 250 Single Grinder-Polisher (Buehler, USA) of 600 Grit to flatten the surface. The rotation speed was 100 rpm, and the size of the abrasive papers was 8 inches.

Next, the enamel surface was etched with an etching solution (Ormco, CA) for 30 seconds and rinsed with a flow of tap water for 10 seconds. The sample was dried using a handheld dryer for 15 seconds. After the sample's surface had been dried, a thin layer of Ortho Solo primer (Ormco, CA) was applied to enhance the bonding. Then, a pea-size of Blüglöo adhesive (Ormco, CA) was applied to the dental brackets (3M Unitek, USA) to be bonded to the enamel surface. The bracket was pressed onto the enamel surface for approximately 1 minute to ensure the thickness of the material is as thin as possible. The estimated thickness of the adhesive material is 0.03 mm to 0.05 mm.

After that, the adhesive material was cured with a LED curing light (TPC, CA) for 20 seconds to harden the material. The light intensity of the curing light was 1250 mW/cm². Bracket debonding process was done after the samples have been left for a week. Debonding plier (3M, USA) was used to debond the brackets. The tip of the debonding plier was placed against mesial and distal edges of bonding base, and the bracket wings were squeezed mesiodistally to pull the bracket from the tooth surface.

2.2 Laser Irradiation

Teeth samples were irradiated with Nd:YAG laser (Spectron Laser System, UK) with a wavelength of

1064 nm. The laser tip is positioned at 3 cm from the enamel surface. The spot diameter of the laser is 3 mm. The pulse rate is set to 2 Hz. A constant and fixed distance is important to ensure a consistent spot size for each ablated area, as shown in Figure 1. In this study, the energies used were 510 mJ, 540 mJ, and 580 mJ. Four samples were subjected to each energy level.

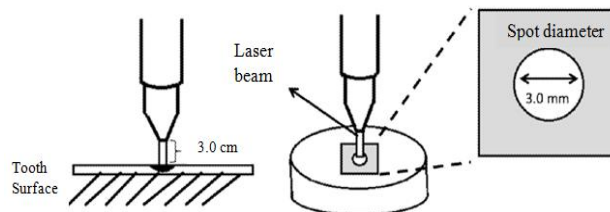


Figure 1 Laser irradiation setting

2.3 Sample Analysis

Samples analysis was done using a Field Emission Scanning Electron Microscope (FESEM) and Electron Dispersive X-ray Spectrometry (EDX). FESEM (FEI Nova NanoSEM 450, USA) was used to analyze the morphological surface of teeth samples visually, while EDX (FEI, USA) analysis was used to analyze the elemental composition of the samples. The morphology and the elemental composition data were compared between samples at the condition before applying the adhesive, after debonding process, and samples after Nd:YAG laser irradiation.

Percentage difference of the elemental composition was also calculated to identify any significant changes in the element when subject to laser irradiation. The percentage difference (PD) is calculated as:

$$PD(\%) = \frac{\text{Element}_{(\text{after})} - \text{Element}_{(\text{before})}}{\text{Element}_{(\text{after})} + \text{Element}_{(\text{before})}} \times 100\%$$

The element is either carbon, oxygen, phosphorus or calcium, depending on the element of interest.

2.4 Statistical Analysis

Groups of data were expressed as means and standard deviation. P-value less than 0.5 is considered statistically significant. A paired samples t-test was used to compare the differences between groups of samples without adhesive, samples after debonding and samples after laser irradiation for each element. Analysis was conducted using Statistical Package for the Social Sciences (SPSS) (IBM Corporation, USA) software.

3.0 RESULTS AND DISCUSSION

3.1 Surface Morphology

Figure 2(a) shows the surface morphology of a human tooth without adhesive material, selected from one of the samples in this study. Additionally, Figure 2(b) shows the human tooth after bracket debonding process. Tooth sample without adhesive showed a smooth and uniform surface. While in Figure 2(b), due to the presence of adhesive, the tooth sample appeared rougher and non-uniform texture can be seen throughout the sample surface.

After Nd:YAG laser irradiation, spots on the samples were captured to assess any different patterns produced on the sample surface. Figure 3 shows the surface morphology of the tooth sample after laser irradiation of 510, 540 and 580 mJ. After irradiated with 510 mJ of energy, the formation of holes is produced in the samples. The holes formation of the sample in Figure 3 are more profound and more extensive compared to the holes created in Figure 3(b). The formation of holes is due to the exposure of laser irradiation on the desired area. The presence of bubbles also can be found in the surrounding exposure area, which defines the melting effect of adhesives has occurred. Both Figure 3(a) and Figure 3(b) show that the adhesive materials were not fully removed during the irradiation process.

Figures 3(c) and 3(d) show the surface morphology of tooth samples after laser irradiation of 540 mJ. Smooth texture for both samples with less indentation on the surface can be seen on the tooth sample after it has been irradiated with 540 mJ. The teeth surface showed a scattered grain pattern, which indicates potential complete removal of the adhesives on the irradiated area. Samples irradiated with 580 mJ of energy produced a surface with non-uniformity and rough texture, as seen in Figure 3(e). Whereas, another sample had holes with amorphous grain surface, as shown in Figure 3(f). Based on the changes found in enamel's microstructures after laser irradiation, the results in this study are similar to the expected outcome.

Enamel surface was expected to undergo few physical changes such as the formation of holes or bubble-like structures, numerous pores, pits and fissures after laser irradiation [19, 20]. A study by Al-Jedani *et al.* [21] showed that the original microstructure of the enamel rod might change and became irregular in shape due to the melting of the surface after laser exposure. Additionally, the appearance of pores and bubbles may also exist [22]. The presence of small shallow cavities surrounds enamel granules are the characteristics found on enamel surface after irradiated with Nd:YAG laser [23].

The resulting structure of the tooth sample highly depends on the irradiation energy exposed to the sample, surface and structure of the target.

Eventhough same laser energy has been exposed to a sample, the results for both laser irradiation may not be the same. Both surfaces produced a different level of depth penetration and variation in enamel's morphological changes after laser exposure. This is due to the difference of the material's thickness on the convex surface of the enamel [13]. This condition influenced the laser penetration level on the material and enamel surface, thus results in varies melting effect in the material removal process.

3.2 Elemental Composition

In this study, four main elements exist in all samples, which are carbon (C), oxygen (O), phosphorus (P) and calcium (Ca). Table 1, 2 and 3 show the elemental composition by weighted percentage (wt.%) and atomic percentage (at.%) for each element that had been identified on the samples without adhesive, samples with adhesive after bracket debonds process and samples after laser irradiation of 510 mJ, 540 mJ and 580 mJ.

In the samples without adhesive, the elemental composition represents the composition of the tooth samples itself without any modifications. At this stage, oxygen is the most abundant element in all samples, followed by calcium. On the other hand, carbon is the least element in all samples. Interestingly, phosphorus and calcium are not detected in the samples after bracket debonds process. Additionally, the percentages of carbon are primarily increased in the samples after bracket debond process. The increased carbon percentage indicates that the tooth surface has been applied with an adhesive material, and a portion of the adhesive material remains on the tooth surface after bracket debond process.

After laser irradiation, carbon is expected to increase due to the ablation process. In this study, it is observed that carbon is higher after laser irradiation compared to the original tooth condition (without adhesive). The percentage of oxygen after irradiation is relatively similar to its original condition. However, the percentage of phosphorus and calcium is varied for all the samples, as shown in Table 1-3.

Table 4 shows the percentage differences of carbon (C), oxygen (O), phosphorus (P) and calcium (Ca) comparing before bracket bonding process and after laser irradiation for laser energy parameters of 510 mJ, 540 mJ, and 580 mJ. The percentage difference of carbon is the highest for samples irradiated with 580 mJ with 72.90 %, while oxygen has the highest percentage difference after irradiated with 510 mJ. Both phosphorus and calcium have the most percentage differences in the energy of 510 mJ, with 97.30 % and 97.83 %, respectively.

For most samples, the weighted percentage of carbon increased in tooth sample with composite adhesives compared to sample without adhesive.

The increasing percentage of carbon is due to the presence of the composite adhesives on the surface. Blügl composite adhesive contains about 10-20 wt.% of bisphenol A diglycidyl ether methacrylate (BADGE), which is a large molecular methacrylate

monomer used in most composite adhesives [24]. The molecular formula of BADGE is $C_{21}H_{24}O_4$, which contributes to the presence of carbon element in the composite adhesives.

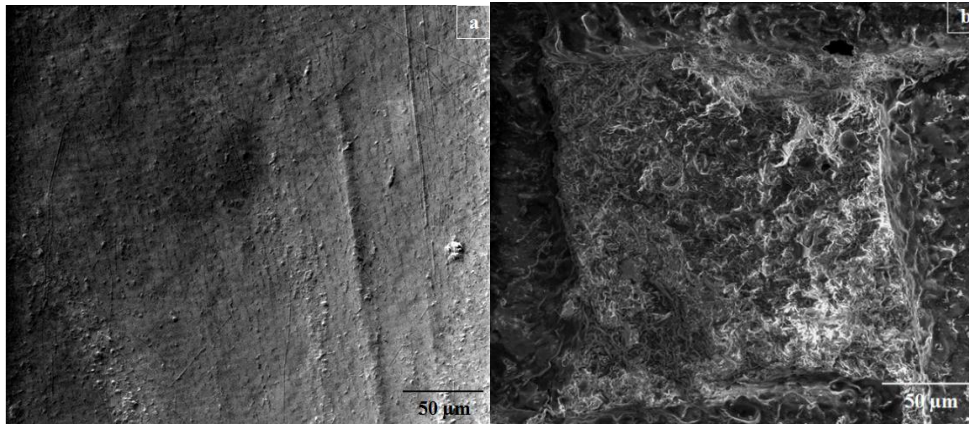


Figure 2 Surface morphology of a tooth (a) without adhesive, and (b) with adhesives after bracket debonding process (1kx magnification)

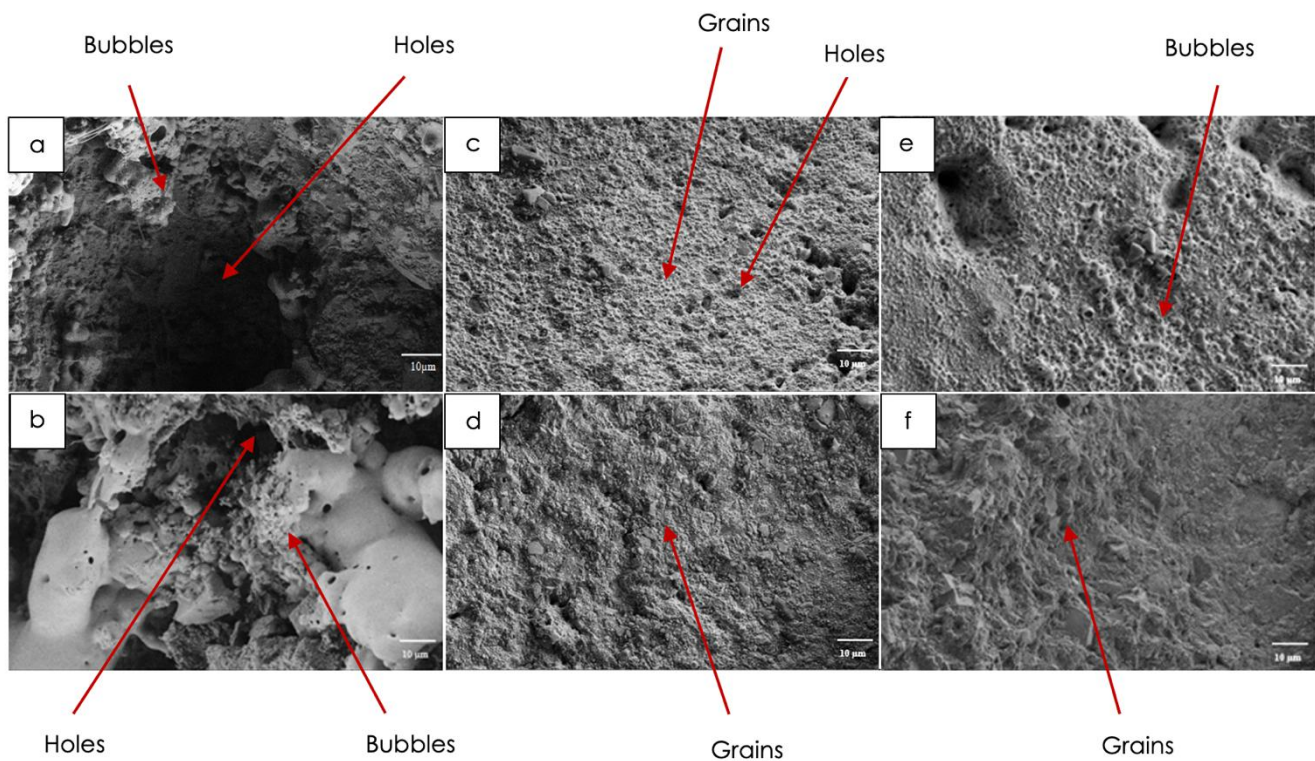


Figure 3 Surface morphology of tooth sample after laser irradiation with energy of 510 mJ (a,b), 540 mJ (c,d), and 580 mJ (e,f) (1kx magnification)

However, after laser irradiation, the carbon percentage in all samples were reduced. The decreasing rate of carbon after laser irradiation indicates that material removal occurred on the

sample. The heating from laser light might cause the enamel to melt on surfaces and eventually reduced a significant amount of carbonate content of hydroxyapatite. This condition makes enamel more

resistant to demineralization [25, 26]. Based on the ablation methodologies concepts, the adhesive absorbs a portion of energy during the absorption process. The adhesives will then become carbonized and led to the removal of the adhesive material from the surface [13].

3.3 Statistical Analysis

Means and standard deviations of carbon, oxygen, phosphorus and calcium were calculated in samples without adhesive, samples with adhesive after bracket debond and samples after laser irradiation. A paired t-test was carried out for each sample to find out the significant difference for each element in the samples. Table 5 shows the statistical analysis of carbon, oxygen, phosphorus and calcium for the samples after debonding compared to the samples without adhesive. Carbon increased significantly with a p-value of 0.002 ($p < 0.05$). Both phosphorus and calcium also increased with p-values of 0.000 and 0.010, respectively.

Table 1 Elemental composition by weighted percentage (wt.%) and atomic percentage (at.%) for each element on the sample without adhesive, after bracket debond and after laser irradiation of 510 mJ

Element	Without Adhesive		After Bracket Debond		After Laser Irradiation	
	wt. %	at. %	wt. %	at. %	wt. %	at. %
C	4.87	8.66	36.54	46.10	5.94	10.10
O	46.88	62.60	47.82	45.29	41.65	53.21
P	17.26	11.90	0.00	0.00	0.23	0.15
Ca	30.14	16.06	0.00	0.00	0.33	0.17

Table 2 Elemental composition by weighted percentage (wt.%) and atomic percentage (at.%) for each element on the sample without adhesive, after bracket debond and after laser irradiation of 540 mJ

Element	Without Adhesive		After Bracket Debond		After Laser Irradiation	
	wt. %	at. %	wt. %	at. %	wt. %	at. %
C	7.99	15.24	40.17	42.47	29.43	30.98
O	33.58	48.05	10.00	25.01	35.94	25.94
P	18.74	13.85	0.00	0.00	6.54	3.66
Ca	38.90	22.22	0.00	0.00	13.11	5.67

Table 3 Elemental composition by weighted percentage (wt.%) and atomic percentage (at.%) for each element on the sample without adhesive, after bracket debond and after laser irradiation of 580 mJ

Element	Without Adhesive		After Bracket Debond		After Laser Irradiation	
	wt. %	at. %	wt. %	at. %	wt. %	at. %
C	5.22	10.23	33.30	43.02	33.12	47.31
O	34.44	50.62	46.64	45.49	32.74	34.92
P	19.33	14.69	0.00	0.00	12.99	3.83
Ca	40.29	23.67	0.00	0.00	6.96	5.80

Table 4 Percentage differences of carbon (C), oxygen (O), phosphorus (P) and calcium (Ca) comparing after laser irradiation and before bracket bonding process for various laser energy

Energy (mJ)	C	O	P	Ca
510	9.90	5.91	97.30	97.83
540	57.28	3.39	48.26	49.59
580	72.90	2.53	19.62	70.54

Additionally, Table 6 shows the statistical analysis comparing the samples after bracket debond and samples after laser irradiation. There were significant changes in carbon, phosphorus, and calcium, where carbon increased with p-values of 0.021, phosphorus with 0.049 and calcium with 0.043.

Table 5 Statistical analysis of carbon, oxygen, calcium and phosphorus for samples without adhesive and after debonding for energy variation

	Without Adhesive Mean (SD)	After Debond Mean (SD)	Mean of Score Difference (95% CI)	T Stat ^a (df)	P Value ^a
C	12.8 (13.2)	42.6 (7.5)	-29.8 (-43.3,-16.2)	-5.4(6)	0.002
O	44.0 (8.2)	38.8 (13.1)	5.2 (-5.5,15.8)	1.2(6)	0.284
P	15.3 (4.0)	0.1 (0.2)	15.2 (11.4,18.9)	9.9(6)	0.000
Ca	22.7 (16.1)	0.0 (0.1)	22.8 (7.8,37.7)	3.7(6)	0.010

^aPaired t-test

Table 6 Statistical analysis of carbon, oxygen, calcium and phosphorus for samples between after debonding and after laser irradiation for energy variation

	After Debond Mean (SD)	After Laser Mean (SD)	Mean of Score Difference (95% CI)	T Stat ^a (df)	P Value ^a
C	42.6 (7.5)	22.7 (16.9)	19.9 (4.1,35.6)	3.1(6)	0.021
O	38.8 (13.1)	34.1 (4.9)	4.7 (-8.1,17.5)	0.9(6)	0.402
P	0.1 (0.2)	6.2 (6.5)	-6.2 (-12.3,0.0)	-2.5(6)	0.049
Ca	0.0 (0.0)	16.7 (17.2)	-16.7 (-32.6,-0.7)	-2.6(6)	0.043

^aPaired t-test

Table 7 shows the statistical analysis of changes in carbon, oxygen, calcium and phosphorus of the samples between after debonding and after laser irradiation based on the energy of the laser. Using multiple post-hoc comparison (Tukey HSD), there was a significant mean difference for carbon between the energy of 580, 540 and 510 mJ ($p < 0.05$). The result indicates a higher mean difference

between the energy of 510 mJ and 580 mJ compared to the energy of 540 mJ and 580 mJ.

Table 7 Statistical analysis of change in carbon, oxygen, calcium and phosphorus of the samples between after debonding and after laser irradiation based on the energy of the laser

	Energy (mJ)	Mean (SD)	(95% CI)	P Value ^a
C	510	-24.3 (58.0)	-72.71, 24.2	<0.001
	540	-12.1 (43.2)	-48.2, 24.0	
	580	14.1 (23.1)	-5.23, 33.37	
O	510	-10.0 (12.2)	-20.2, 0.3	0.303
	540	-20.2 (25.9)	-41.9, 1.4	
	580	-20.3 (10.9)	-29.3, -11.2	
P	510	-50.0 (53.6)	-94.9, -5.2	0.344
	540	-45.2 (58.7)	-94.3, 3.9	
	580	-47.9 (55.9)	-94.6, -1.1	
Ca	510	-36.6 (53.9)	-81.6, 8.5	0.578
	540	-40.4 (64.3)	-94.2, 13.3	
	580	-48.4 (55.3)	-94.6, -2.2	

^aMultiple post-hoc comparison (Tukey HSD)

4.0 CONCLUSION

Several morphological changes occurred on the enamel surface after samples were irradiated with various laser energy parameters. Based on the results, the effectiveness of material removal on the enamel surface highly depends on the irradiation energy level. As seen from this study, 510 mJ is insufficient to remove the adhesive material. However, 540 mJ of energy can potentially be used for this purpose. Additionally, other factors such as the thickness of the adhesive material, and adhesive material composition applied on the tooth surface may also influence the material removal process. Further investigation is required to study the effect of higher energy and ablation concepts on the enamel surface.

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