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- Betel leaf toothpastes inhibit dental plaque formation on fixed orthodontic patients
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Research Report

In vitro effect of Q-switched Nd:YAG laser exposure on morphology, hydroxyapatite composition and microhardness properties of human dentin

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ABSTRACT

Background: A Q-switched Nd:YAG laser was employed as a source of ablation. The fundamental wavelength of the laser is 1064 nm, with pulse duration of 8 nanosecond operates with uniphase mode of TEM₀₀. In the following experiments, dentin samples (without caries and plaque) are exposed to pulse laser with Q-switching effect at various energy dose. **Purpose:** The aim of this study was to investigate the effect of laser ablation on dentin samples using Q-switched Nd:YAG laser exposure. **Methods:** The laser was operated in repetitive mode with frequency of 10 Hz. The energy dose of the laser was ranging from 13.9 J/cm², 21.2 J/cm² and 41.7 J/cm². The target material comprised of human dentin. The laser was exposed in one mode with Q-switched Nd:YAG laser. Energy delivered to the target through free beam technique. The exposed human dentin was examined by using x-ray diffraction (XRD) and fluorescence scanning electron microscopy for energy dispersive (FESEM-EDAX). Microhardness of human dentin were examined by using microhardness vickers test (MVT). **Results:** The result obtained showed that the composition of hydroxyapatite of the dentin after exposed by Q-switched Nd:YAG laser are 75.02% to 78.21%, with microhardness of 38.7 kgf/mm² to 86.6 kgf/mm². This indicated that exposed pulsed Nd:YAG laser on the human dentin attributed to the photothermal effect. The power density created by the Q-switched Nd:YAG laser enables the heat to produce optical breakdown (melting and hole) associated with plasma formation and shock wave propagation, from energy dose of 21.2 J/cm². From XRD analysis showed that the exposure of Nd:YAG laser did not involve in changing the crystal structure of the dentin, but due to photoablation effect. **Conclusion:** In conclusion, the application of Q-switched Nd:YAG laser as contactless drills in dentistry should be regarded as an alternative to the classical mechanical technique to improve the quality of the dentin treatment.

Key words: Plasma, laser ablation, crystal structure, microhardness, hydroxyapatite, Nd:YAG laser

ABSTRAK

Latar belakang: Mode Q-Switch pada laser Nd:YAG dapat menghasilkan fenomena ablasi pada dentin. Laser Nd:YAG yang digunakan mempunyai panjang gelombang 1064 nm, durasi pulsa 8 ns beroperasi dengan mode TEM₀₀. Sampel dentin yang digunakan tanpa karies dan plak, yang dipapari laser dengan mode Q-switch dalam berbagai variasi dosis energi. **Tujuan:** Tujuan penelitian ini adalah mengamati efek ablasi dentin secara in vitro akibat paparan laser Nd:YAG Q-switch dengan pengamatan morfologi permukaan, komposisi hidroksiapatit, dan uji kekerasan mikro. **Metode:** Laser Nd:YAG Q-switch dengan frekuensi 10 Hz dan variasi dosis energi 13,9 J/cm², 21,2 J/cm² dan 41,7 J/cm² ditembakkan pada sampel dentin manusia dengan teknik penyinaran bebas tanpa dilewatkan serat optik. Dentin yang terbuka diamati menggunakan x-ray diffraction (XRD) dan fluorescence scanning electron microscopy for energy dispersive (FESEM-EDAX). Kekerasan mikro dari dentin juga diamati menggunakan microhardness vickers test (MVT). **Hasil:** Hasil penelitian menunjukkan bahwa komposisi hidroksiapatit dari dentin setelah paparan laser Nd:YAG Q-Switch menunjukkan peningkatan berkisar 75,02% sampai 78,21% dibandingkan normal, dengan kekerasan mikro berkisar 38,7 kgf/mm² sampai 86,6 kgf/mm². Perubahan pada struktur mikro tersebut disebabkan karena adanya efek fototermal. Kerapatan daya yang bervariasi berdasarkan variasi dosis energi menyebabkan efek panas pada dentin yang menyebabkan adanya fenomena optical breakdown, yang ditandai dengan munculnya efek leleh dan lubang pada

sampel karena produksi plasma dan adanya gelombang kejut, mulai dosis 21,2 J/cm². Berdasarkan uji XRD, efek yang muncul pada dentin tidak menyebabkan terjadinya perubahan struktur kristal hidroksiapatit, tetapi menyebabkan perubahan komposisi hidroksiapatit yang disebut dengan fotoablasi. **Kesimpulan:** Dapat disimpulkan bahwa penggunaan mode Q-switched pada laser Nd:YAG sebagai alat dengan kontak minimal dapat dijadikan teknik alternatif untuk meningkatkan kualitas perawatan dental.

Kata kunci: Plasma, ablasi laser, struktur kristal, kekerasan mikro, hidroksiapatit, laser Nd:YAG

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INTRODUCTION

Nd:YAG laser technology has continuously developed during the last years partly driven by the medical demand or adapted from technical application and transferred to the medical use, especially in dentistry. Laser has firmly established itself as an indispensable tool for various applications such as in micromachining and medical surgery.^{1–5} Dental laser based on Nd:YAG laser is used in dentistry area for therapy supporting on soft tissues in 1980. Recently, laser development is used to save application for dental hard tissues therapy. In dentistry, laser is used for supporting therapy on soft tissues and evaluating dental enamel, caries morphology, dentine resistance, and dental plaque composition.^{1,4–6} Among the most used laser systems in dentistry relies one Nd:YAG laser with a wavelength of 1064 nm assuring the specific characteristic of presenting affinity with pigmented tissues, which particularly makes it selective for carious tissues.^{1,2} Nd:YAG laser (1064 nm) is also used in cosmetic application,³ photomedicine and laser surgery.^{4,5} Nd:YAG laser irradiated was used to evaluate the dental enamel caries morphology through SEM images,¹ dental plaque composition of human enamel,⁴ surface dentin modification,^{8,9} photothermal and optical breakdown production.¹⁰

The aim of this in vitro study was to investigate the effect of a Q-switched Nd:YAG laser irradiated to healthy human dentin (without caries and plaque) by regulating the voltage oscillator ranging from 660 to 740 Volt, with the variation of power density are 13.9 J/cm², 21.2 J/cm² and 41.7 J/cm². There are three effects of human dentin that will be measured: surface morphology through fluorescence scanning electron microscopy for energy dispersive x-ray (FESEM-EDAX) images, hydroxyapatite composition through x-ray diffraction (XRD) analysis, and microhardness properties.

MATERIALS AND METHODS

A Q-switched Nd:YAG laser with fundamental wavelength of 1064 nm manufactured by Lumonics model HY 200, was employed as a source of ablation. The pulse duration of the beam is 8 nanoseconds and operates with uniphase mode of TEM₀₀. The laser was conducted in

repetitive mode with frequency of 10 Hz. The various energy dose of the laser are 13.9 J/cm², 21.2 J/cm² and 41.7 J/cm². The energy of Nd:YAG laser was verified by regulating the capacitor voltage in xenon flashlamp driver ranging from 640 to 740 Volt.

The specimens comprised of healthy human dentin (upper premolar) without caries and plaque. The samples provided by Oral Surgery Clinic, Faculty of Dentistry, Airlangga University. The teeth were extracted from consenting patients (ages of 13 until 19 years old) who undergoing extractions for orthodontic treatment. Prior to teeth collection, approvals for using human hard tissue samples were obtained from the human ethics committees of all participating institutes involved in this study. The samples were cleaned using Ultrasonicator Branson 5210 at normal temperature within 10 minutes duration. The cleaned teeth were then stored in distilled water at constant temperature of 4° C before ready to cut. The purpose of cutting is to obtain a flat dentin surface. Machine model of Edenta with diamond disk was employed to cut the sample at low speed. The dentin surfaces were polished using abrasive paper grading from 400 to 2000 with diameter ranging of 1.5 to 2 mm. After polishing, the specimens were randomly divided into five groups according to the class of delivering energy.

The Nd:YAG laser was conducted in one modes with Q-switched. The laser was focused using converging lens of 28 mm focal length. Protective goggles specific for this wavelength for eye safety and also double masks were used to avoid vapor aspiration during the irradiation procedure. The exposed human dentin was examined by metallurgical method. In this case XRD (Siemens Diffractometer D 5000) and FESEM-EDAX (Zeiss Supra 35 VP) machines were conducted. Microhardness properties of human dentin were examined by using MVT (Future Tech FM 7). The surface of specimen was stressed by 2 kgf using piramide diamond in this system which inclined at an angle of 136°.

The optical alignment of this experimental set-up is shown in figure 1. Distance between human enamel and beam splitter is 50 cm, and exposure time was set around 9–11 second. He-Ne laser was also coaxial with the Nd:YAG laser for an ease of alignment, because Nd:YAG laser produced invisible light. Couple charge device video camera also, utilized for an accurate and precisely location of the sample.

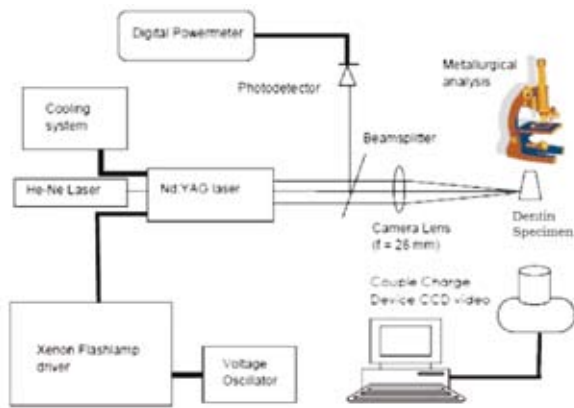


Figure 1. Experimental set up to expose human dentin with Q-Switched Nd:YAG laser.

RESULTS

The exposed and unexposed dentin specimens were observed under FESEM. The typical results are shown in figure 2. Initially the unexposed of dentin surface was observed. The morphological of normal dentin is depicted in figure 2a. The exposed dentin with Q-switched Nd:YAG laser using energy dose of 13.9 J/cm^2 , operated pumping voltage at 660 V, and repetition rate of 10 Hz is depicted in

figure 2b. Entirely different structure of damage occurred when the dentin surface was focused by a Q-switched Nd:YAG laser such as shown in figure 2c using energy dose of 21.2 J/cm^2 , operated pumping voltage at 700 V, and repetition rate of 10 Hz, and figure 2d using energy dose 41.7 J/cm^2 , operated pumping voltage at 740 V, and repetition rate of 10 Hz.

The typical of XRD results are emerging in figure 3-a for normal human dentin, figure 3b for Q-Switched Nd:YAG laser operated pumping voltage at 660 V with energy dose and repetitive rate of 10 Hz, and figure 3c for Q-Switched Nd:YAG laser using pumping voltage of 700 V, energy dose of 21.2 J/cm^2 and frequency of 10 Hz, and figure 3d for Q-switched Nd:YAG laser using pumping voltage of 740 V, energy dose of 41.7 J/cm^2 and frequency of 10 Hz. The peaks in each figure represented the hydroxyapatite compound $[\text{Ca}_{10}(\text{PO}_4)_6](\text{OH})_2$ of the tested human dentin sample. The percentage of the composition for each tested sample are calculated based on this figure 3. The computed results are tabulated in table 1. The Lattice parameters for each tested sample are also shown in the this table.

The results of microhardness measurement of the human dentin after the exposure to the pulsed laser with and without Q-switching are tabulated in table 2. At a maximum energy dose of 41.7 J/cm^2 , the microhardness of the dentin is measured to be 86.6 kgf/mm^2 with Q-switched Nd:YAG laser.

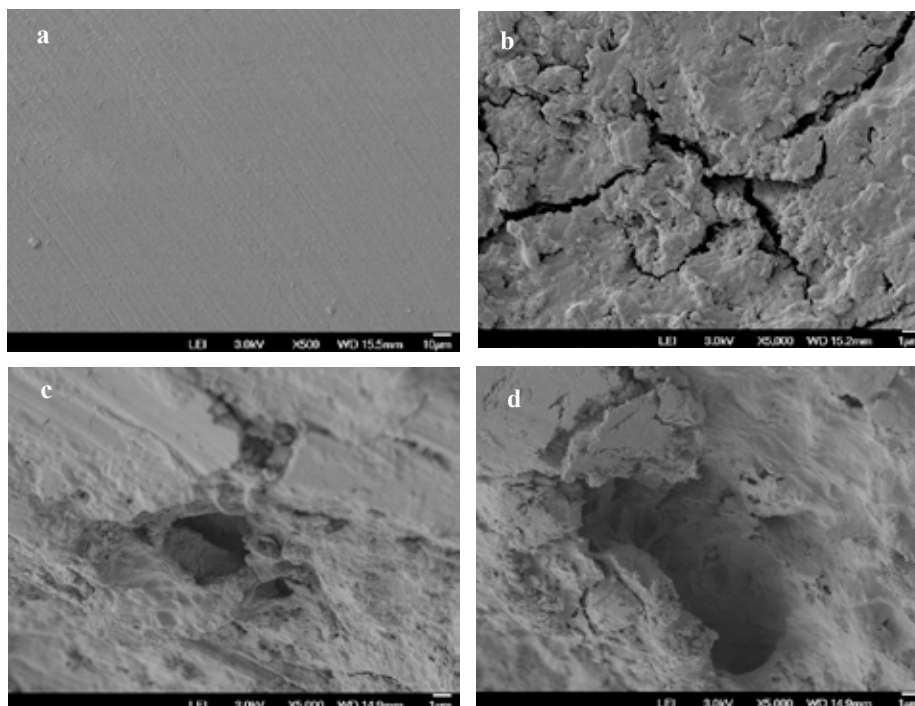


Figure 2. Enamel surface morphology using FESEM and repetition rate of 10 Hz. a) without exposure (normal human dentin); b) with exposure to energy dose of 13.9 J/cm^2 , operated pumping voltage at 660 V); c) with exposure to energy dose of 21.2 J/cm^2 , operated pumping voltage at 700 V); d) with exposure to energy dose of 41.7 J/cm^2 , operated pumping voltage at 740 V).

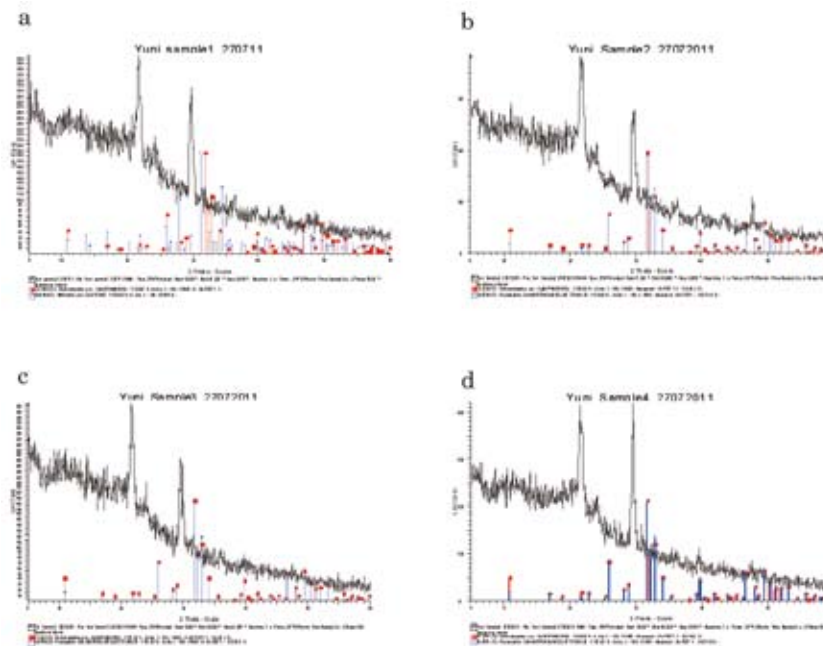


Figure 3. XRD spectra of the dentin specimens at energy dose various and fixed repetition rate of 10 Hz
 a) XRD output from normal dentin; b) XRD output of dentin exposed to *Q*-switched Nd:YAG laser (energy dose of 13.9 J/cm²); c) XRD output of dentin exposed to *Q*-switched Nd:YAG laser (energy dose of 21.2 J/cm²); d) XRD output of dentin exposed to *Q*-switched Nd:YAG laser (energy dose of 41.7 J/cm²).

Table 1. Percentage of hydroxyapatite compound [Ca₁₀(PO₄)₆(OH)₂] using *Q*-switched Nd:YAG laser with repetition rate of 10 Hz

No	Energy dose (J/cm ²)	Lattice parameter (Å)	Percentage of hydroxyapatite (%)
1	0	a=b=9.416; c=6.884	73.42
2	13.9	a=b=9.399; c=6.883	75.02
3	21.2	a=b=9.463; c=6.856	76.03
4	41.9	a=b=9.447; c=6.832	78.21

Table 2. Microhardness properties of human dentin samples with *Q*-switched Nd:YAG laser exposure

No	Energy dose (J/cm ²)	Hardness value (kgf/mm ²)
1	0	32.3
2	13.9	38.7
3	21.2	46.9
4	41.9	86.6

DISCUSSION

There were many microcracking observed on the dentin surface exposed to laser with the *Q*-switching on energy dose of 13.9 J/cm². The surface comprised of microcrack

all over the surface. The focused of Nd:YAG pulsed laser acts like hammering. The impact results rough surface associated with microcrack. This means the infrared of Nd:YAG laser produced thermal effect which absorbed the surface and diffused the heat laterally. The multiple impacts of the focused pulsed laser cause the surface dehydrated and crack. This is attributed to excess heat, which causes surface dehydration and crack. In this case, the multiple impacts of the focused pulsed laser cause rough heat diffusion, which affects the interaction between light and hard tissue. However, an entirely different structural damage was observed when the dentin surface was exposed to a *Q*-switched Nd:YAG laser, as shown in figure 2c on energy dose of 21.2 J/cm² and figure 2d on energy dose of 41.7 J/cm², that occurred melting and variation of deep hole. Although the dentin exposed by multiple impacts of pulse laser but only one pit is appeared on the dentin surface. The pit like a deep hole been drilled by the focused laser. Power density created by the *Q*-switched Nd:YAG laser enable to produce breakdown associated with plasma formation and shock wave propagation. The high temperature from the plasma and high pressure from the shock wave responsible to melt and vaporize the enamel surface which result the formation of deep hole, depending on variation energy dose.

Furthermore, damage observed under FESEM analysis due to *Q*-switched exposed is distributed more uniformly as a resulted of mechanical mechanism when sample preparation. Rather than only single deep pit due to the effect of melted and vaporization after interaction with

microplasma induced by focused of Q-switched Nd:YAG laser, associated with pressure wave induced by the propagation of acoustic shock wave. Non-switched is more energetic in this interaction subjected to higher energy carried by every single pulse, compared to Q-switched laser. Furthermore, the longer duration and the integration of pulsed energy delivered by multipulses on the same spot, subject to greater and longer exposure to the surface of human dentin. In contrast with Q-switched the peak power deliver to the target is much higher but the duration within nanosecond time will not allow the thermal conductivity on surface of human dentin. This result is more localized, damage the pits, and may cause some particle removal due to the melted and vaporization effect.

In this experiment, the Q-switched Nd:YAG lasers operate at energi dose of 13.9 J/cm^2 , 21.2 J/cm^2 , and 41.7 J/cm^2 with repetitive rates of 10 Hz. Hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) is found having high percentage after exposed by Q-switched Nd:YAG laser. These results show that human dentin consists of more than 73% of hydroxyapatite, in which the ablation threshold fluence is expected to be. It is lower than that of monocrystalline fluoroapatite. The composition of hydroxyapatite of the dentin after exposure to Q-switched Nd:YAG laser varies from 75.02% to 78.21%. This results indicate that exposing pulsed Nd:YAG laser on the human dentin induced greater composition of hydroxyapatite. The chemical bonding between the atoms in molecules are affected due to the vibration or rotational results from the absorption of infrared laser radiation. This effect of chemical composition is also indicated by the shifted on the lattice parameter of the crystal structure of the dentin material. After the dentin specimen exposed by Q-switched infrared laser, the lattice parameters of the hydroxyapatite is shifted greater than compared to the exposure by normal dentin unexposed as long as no new appearance of peaks of loss of peaks meaning that the crystal structure of the dentin material is remain the same. The results also proved by the XRD spectrum analysis (Figure 3). This indicated that exposed by pulsed Nd:YAG laser on the human dentin induced greater composition of hydroxyapatite and higher microhardness in comparison to normal unexposed dentin. The absorption of infrared laser radiation also affects the chemical bonding between the atoms in molecules due to the vibration or rotational effect. This resulted in a shift of lattice parameter of the crystal structure of the dentin material. It is also observed that the Q-switched pulses exposure shifts the lattice parameters of the hydroxyapatite greater than that of the non-Q-switched laser. Since no new appearance of peaks of loss is observed in the XRD spectra the crystal structure of the dentin materials assumed unchanged. Exposure of pulsed Nd:YAG laser on the human dentin caused photoablation effect.

Exposed the dentin by infrared laser radiation is similar as laser annealing process. The longer the time taken to

anneal the surface the more the harder the surface due to the loss of water and carbon content of the dentin material. The results of the microhardness properties of the dentin after anneal with infrared material is shown in table 2. This means the pulse Nd:YAG laser exposed almost 15000 times longer than nanosecond signal from Q-switched Nd:YAG laser. Furthermore the power received after 10 second exposure for example by the dentin surface is almost 166 W for 10 Hz repetition rate. Such power radiation will be enough to generate high temperature within 100 to 650°C since the area of the enamel just around 4 to 9 mm in diameter. The photothermal effect is possible to decrease the water and carbon content from the dentin surface. The evaporation of water and carbon content is subjected to increase the microhardness property of the dentin. This result is in good agreement with other previous researchers.^{1,4,6-9}

These results show that nanoseconds Q-switched Nd:YAG laser can be used for tooth ablation applications. The material removal remains localized on the area of the laser spot. Almost no signs of collateral damage and cracks are observed with a sharply defined cavity edges. This study suggests that the application of short-pulse lasers as contactless drills in dentistry can be considered as an alternative to the classical mechanical technique to improve the quality of the dentin treatment.

Ablation of the dentin is demonstrated at localized area and no signs of collateral damage and cracks are observed using an 8 ns pulses at wavelength of 1064 nm, energy dose of 13.9 J/cm^2 and 10 Hz repetition rate from a Q-switched Nd:YAG laser, while the energy dose of 21.2 J/cm^2 and 41.7 J/cm^2 occurred melting and deep hole. This exposure from Q-switched Nd:YAG laser increases the microhardness property and hydroapatite percentage of the dentin, which produces a side effect of surface dehydration and crack, melting, and deep hole. This side effect can use to dentin treatment in dentistry. However, the crystal structure of dentin is unchanged since the shifted crystal lattice is small with exposure to Q-switched Nd:YAG laser, so safe for application to dentin treatment. In conclusion the application of Q-switched Nd:YAG laser as contactless drills in dentistry should be regarded as an alternative to the classical mechanical technique to improve the quality of the dentin treatment.

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