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ORIGINAL ARTICLE

Wettability of dentin after Yb:KYW thin-disk femtosecond ablation

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Abstract The aim of this study was to quantitatively evaluate the wettability of dentin after Yb:KYW thin-disk femtosecond-pulsed laser ablation by measuring the contact angle. Different laser parameters were used including different fluences (*F*), scanning speeds, and scanning line spacings. Crowns of 15 extracted human teeth were cut longitudinally into slices approximately 1.5-mm thick with a cutting instrument. The samples were randomly divided into ten groups (*n* =3/group). Samples in groups 1–8 were irradiated with a femtosecond-pulsed laser. The dentin samples were fixed on a stage at the focal plane, and the laser beam irradiated the samples through a galvanometric scanning system so rectangular movement could be achieved. Samples in groups 9 and 10 were prepared with grinding instruments. Following ablation and preparation, the samples were examined for contact angle with an optical contact angle measuring instrument. The results showed that scanning speed and scanning line spacing had little influence on the wettability of dentin following femtosecond-pulsed laser ablation, except when F=6 J/cm^2. For six out of the eight laser ablation groups, when a lower fluence was used, the dentin contact angle was higher and vice versa. Most of the dentin which had been ablated using the femtosecond-pulsed laser had improved wettability compared to samples prepared with the grinding instruments. This study showed that various laser fluences, scanning speeds, and scanning line spacings can alter dentin wettability. Therefore, adequate parameters should be chosen to achieve proper therapeutic benefits.

Keywords Femtosecond laser · Dentin · Wettability · ?Contact angle

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Introduction

Since the bond between tooth structure and restorative materials significantly influences the quality and longevity of restorative treatment, this bond is a critical factor in the overall clinical outcome for dental patients. Much work has been done with the goal of obtaining a permanent adhesion between the tooth and the restorative materials. Generally, three major factors are necessary to ensure the proper formation and permanence of an adhesive bond: (1) establishing intimate contact between the liquid adhesive and the solid adherend, i.e., good wetting must take place; (2) minimizing the stress concentrated at the interface; and (3) minimizing attack on the interface by environmental factors [1, 2]. The majority of researchers agree that the wettability of the enamel and dentin surface is the most important factor for enhancing the adhesion of restorative materials used in dentistry [3]. There are three major factors that influence the wetting of a solid surface by a liquid [1]. The first factor is the surface free energy of the solid and the liquid. This is a function of the chemical composition and heterogeneity of both the liquid and the solid surfaces. For example, the formation of the smear layer after

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grinding the tooth would influence the wetting process and would subsequently affect the bond [4]. The second factor is the surface topography of the adherend. It is presumed that surface rugosity and irregularities increase wettability by increasing the surface area available for bonding. However, excessive surface roughness may lead to difficulty in the flow of the liquid adhesive and thus produce air pockets at the interface [5]. The third factor influencing the wettability is the viscosity of the liquid itself [6].

In recent years, the introduction of lasers into the field as a potential tool for oral medicine has attracted much attention [7–14]. Some investigators have shown that dental hard tissue could be precisely ablated with a femtosecond-pulsed laser; with appropriate laser and scanning parameters, collateral damage involved could be minimal [15–23]. In previous reports, it has been indicated that femtosecond-pulsed laser use alters the surface roughness of dental hard tissues, depending on the parameters used [24, 25]. Since surface roughness is a major factor that influences the wetting of a solid surface by a liquid; the purpose of this study is to evaluate the wettability of femtosecond-ablated dental hard tissues and provide a basis for the usage of femtosecond-pulsed lasers in oral clinical treatments.

Materials and methods

Sample preparation

Fifteen extracted human third molars were used in this study. The teeth were soaked in formalin solution for 2 weeks after extraction and sectioned into crowns and roots along the cementum-enamel junction. The crowns were then cut longitudinally into 30 slices of approximately 1.5 mm in thickness with a cutting instrument (STX202, KEJING, Shenyang, China). Subsequently, the dentin sheets were ground with 600-, 800-, and 1200-grit sandpaper and randomly divided into ten groups of three. The samples were then stored in saline solution to prevent them from drying out until the irradiation began.

Laser ablation

Irradiation was performed with a Yb:KYW diode-pumped solid-state thin-disk femtosecond laser (JenLas D2.fs, Jena, Germany), which produces a wavelength of 1025 nm and pulses of less than 400 fs. This laser system generates an output power of up to 4 W and pulse repetition rate (f) of 30–200 kHz. A pulse repetition rate of 100 kHz was used in this study. The laser beam was focused on the sample surfaces through a galvanometric scanning system (GO2-YAG-12-22-D, Beijing JCZ Technology Co., Ltd., Beijing, China) with a focus distance of 100 mm and a focal spot diameter (φ) of approximately 24 μm. Eight groups (G1–G8) of samples were treated with the laser, and the parameters used are described in Table 1.

The dental samples were fixed on a stage in the focal plane, and parallel lines were irradiated on samples at different fluences (F), scanning speeds (v), and scanning line spacings (s). In this study, the scanning speed determined the pulse overlap within a scanning line, while the scanning line spacing determined the pulse overlap between two scanning lines. The pulse overlaps can be described as: pulse overlap within a scanning line=$(\varphi-v/f)/\varphi$ and pulse overlap between two scanning lines=$(\varphi-s)/\varphi$. In this study, when v=2400 mm/s, s=24 μm; similarly, when v=1200 mm/s, s=12 μm. This ensures that the pulse overlaps within and between scanning lines are the same. Specific fluences were obtained by tuning the output power.

Mechanical preparation

The remaining two groups (G9 and G10) were used as the wettability control. A turbine handpiece (BORALINA, Bien-Air, Bienne, Switzerland) was used at a speed of 310,000 rpm. G9 samples were ground with a diamond bur (TF-12, MANI, Japan), and G10 samples were ground with a different diamond bur (FO-20EF, MANI, Japan).

Contact angle measurement

The contact angle was assessed as a function of wettability. An optical contact angle measuring device (OCA15EC, DataPhysics, Germany) and sessile drop technique were used. A 1-μL drop of deionized water was placed on each slightly dried sample surface as the base line for each condition. The side view of the liquid drop was captured with a video camera. Both the left and right contact angles were recorded and each specimen was measured three times. Statistical analysis of all data obtained was performed using SPSS 13.0 for Windows. One-way ANOVA or Student's t test were used to evaluate the

Table 1 Laser parameters used

Group	Fluence (J/cm^2)	Scanning speed (mm/s)	Scanning line spacing (μm)
G1	2	2400	24
G2	4	2400	24
G3	6	2400	24
G4	8	2400	24
G5	2	1200	12
G6	4	1200	12
G7	6	1200	12
G8	8	1200	12

influence of laser fluence, scanning speed, and scanning line spacing on dentin wettability.

Results

The influence of laser fluence, scanning speed, and scanning line spacing on femtosecond-pulsed laser ablated dentin wettability were studied quantitatively. Mean dentin contact angle and standard deviations of each group are described in Table 2 and Fig. 1. The representative views of the liquid drop on laser-ablated dentin and bur-ground dentin are shown in Fig. 2.

One-way ANOVA was carried out within G1 to G4 and G5 to G8. For G1 to G4, we found that G1 was significantly different from the other three groups ($P<0.05$). No significant differences were found between the other three groups ($P>0.05$). For G5 to G8, all of the groups were significantly different from each other ($P<0.05$) except when comparing G5 to G6. When comparing the femtosecond-ablated dentin contact angle with the same fluence but different scanning speed and scanning line spacing, the *t* test revealed that no significant differences existed except when $F=6$ J/cm^2 ($P<0.05$).

Compared to the contact angle of dentin samples prepared using the diamond bur, when $v=2400$ mm/s and $s=24$ μm, G9 was significantly different from all femtosecond laser-ablated groups except when $F=2$ J/cm^2. G10 was significantly different from G4 ($P<0.05$). When $v=1200$ and $s=12$ μm, G9 and G10 were significantly different from G7 and G8 ($P<0.05$). No significant difference existed between G9 and G10 ($P>0.05$).

Discussion

In this study, human dentin was ablated with a Yb:KYW femtosecond-pulsed laser using different parameters. The Yb^{3+}-doped potassium tungstate $KY(WO_4)_2$ (KYW) is of great interest for diode-pumped solid-state lasers in recent years. It has a smaller quantum defect and higher absorption and emission cross sections. Furthermore, Yb:KYW has a broad emission bandwidth, which allows for the generation and amplification of ultrashort pulses [26]. In 2005, M. Strassl et.al. ablated dentin with a Yb:KYW thin-disk laser; the cavities generated in dentin showed a high quality [27]. With the high repetition rate, this kind of laser can improve the ablation efficiency when interacting with dental hard tissues so that the time needed for laser treatment could be shortened. For these reasons, Yb:KYW laser has one of the highest potentials to be one of the future dentistry treatment tools. The wettability of dentin was analyzed quantitatively by measuring the contact angle. Quantitative comparisons between femtosecond pulse laser-ablated dentin and bur-ground dentin were performed.

Table 2 Mean dentin contact angle and standard deviations for each group

Group	Mean (°)	Standard deviation (°)
G1	70.47	10.27
G2	53.98	8.12
G3	55.22	12.44
G4	47.91	19.43
G5	64.23	8.44
G6	61.47	4.78
G7	39.63	1.84
G8	48.13	1.16
G9	68.16	10.36
G10	61.15	8.59

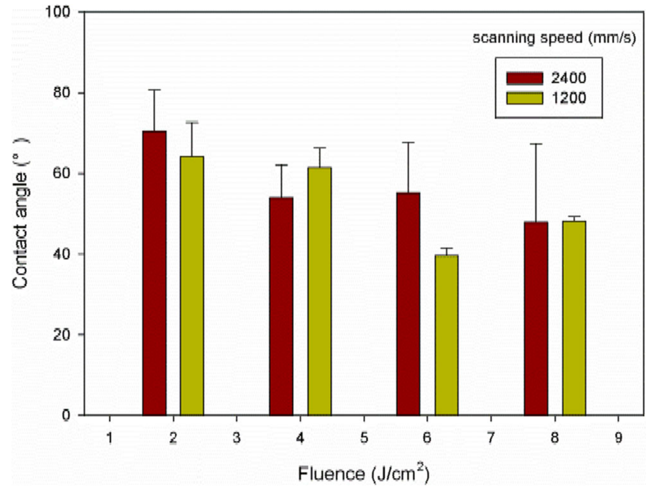

Fig. 1 Contact angle of dentin irradiated with a femtosecond-pulsed laser

Wettability is an important factor for enhancing the bonding between the dental substrate and restorative materials [28, 29]. Contact angle measurement is considered to be a useful indicator of interfacial tension because it has been used as a means of characterizing the wetting of a substrate by the fluid phase [5, 30].

In this study, we found that the parameters of femtosecond-pulsed laser could have an influence on the wettability of dentin. When $v=2400$ mm/s, dentin ablated with the femtosecond-pulsed laser with $F=2$ J/cm^2 had a much higher contact angle compared to dentin ablated with the femtosecond-pulsed laser with higher fluence. We detected the overall trend that the dentin contact angle decreased with increasing laser fluence. The femtosecond-pulsed laser with the lowest fluence used in this study produced the highest contact angle, while the laser with the highest fluence produced the lowest dentin contact angle. However, there were no significant differences found between the three other groups. This overall trend also held true when $v=1200$ mm/s, except

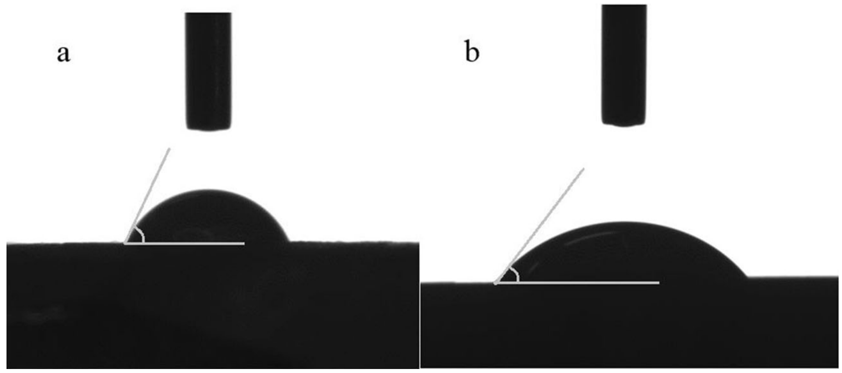

Fig. 2 **a** Contact angle of dentin ground with an FO-20EF diamond bur, contact angle=61.29 °. **b** Contact angle of dentin irradiated with a femtosecond-pulsed laser, F=8 J/cm^2, v=2400 mm/s, s=24 μm, contact angle=48.44 °

when F=8 J/cm^2. It was hypothesized that surface roughness may improve wettability by reducing the contact angle [31]; however, while the roughest dentin surface in our previous study [25] was obtained with ablation from the femtosecond-pulsed laser when F=2 J/cm^2, v=2400 mm/s, and s=24 μm, these inputs resulted in the largest contact angle in this study. This was consistent with some scholars who have stated that roughness has no significant influence on surface wettability [5, 32]. When comparing the contact angle results of dentin samples ablated with the femtosecond-pulsed laser with the same fluence but different scanning speed and scanning line spacing, no significant differences were found except when F =6 J/cm^2. We found that the scanning speed and scanning line spacing had no significant influence on dentin wettability; however, the pulse overlap within and between scanning lines was set to be the same. Further research must be carried out to evaluate how scanning speed and scanning line spacing each influence the dentin wettability.

When compared to the wettability of diamond bur-prepared dentin samples, most of the femtosecond-pulsed laser-ablated dentin samples had a lower contact angle. This may be due to the smear layer that mechanical tools produce [33, 34]. Pashley et al. reported that the smear layer morphology and thickness may have a more important role in influencing the wettability than surface roughness [35] as the smear layer tends to act as a physical barrier to the diffusion of oral fluids and restorative materials to the pulp [5, 35]. It can also occlude the dentinal tubules, thereby reducing the dentin permeability. Debris produced by mechanical tools deposited in the dentinal tubule apertures will prevent fluid from flowing outward onto the surface. The resulting excessive dryness may interfere with bonding between hydrophilic wetting agents and dentin. Therefore, some researchers have recommended the removal of the smear layer to improve bonding [36]. The most commonly used technique to accomplish removal of the smear layer is etching [37]. Dentin ablated with a femtosecond-pulsed laser shows a morphology in which most dentinal tubules remain open [25], and therefore, the outward flow of fluid to the surface could be increased, improving dentin wettability.

Conclusions

Fluence of a femtosecond-pulsed laser had an effect on the wettability of ablated dentin, while scanning speed and scanning line spacing had no significant effect in this study. With the parameters used in this study, a higher fluence appeared to produce a dentin surface with higher contact angle, and lower fluence seemed to lead to improved dentin wettability. Most femtosecond-pulsed laser-ablated dentin samples had improved wettability compared to the samples prepared using mechanical instruments. Adequate parameters should be chosen to achieve the proper therapeutic benefits.

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