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Monitoring Femtosecond Laser Ablation Processes on Human Teeth Using FT-IR Spectroscopy †

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Abstract

In recent years, the laser-based ablation of damaged or undesired tooth material has emerged as a highly promising technique for improving dental cavity preparation. While X-ray diffraction and X-ray photoelectron spectroscopy are generally used to characterize the constituents of ablated surfaces, Fourier Transform Infrared (FT-IR) can also be employed for monitoring the changes induced by the femtosecond laser ablation process. In the present study, FT-IR spectroscopy has been adopted to characterize the changes induced in extracted human teeth. The laser ablation was performed in ambient air by using a femtosecond laser source at different fluences in the range of 0.7–1.5 J/cm^2 to produce regular lines on various samples. Micro-ATR spectroscopy was employed to examine the laser-processed tooth disks. The spectra acquired from different samples reveal the contributions of the various dental components and provide insight into the effect of laser processing under different conditions.

Keywords: femtosecond laser; ablation processes; dentin; enamel; FT-IR spectroscopy

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1. Introduction

The use of lasers in dentistry has long been investigated as an alternative to traditional mechanical drilling, offering the potential for reduced vibration, less noise, and higher precision. Various laser systems have been employed to process hard dental tissues, (enamel and dentin), but the outcomes are highly dependent on the chosen laser parameters. Among these, the pulse duration has emerged as the most critical factor governing the light-tissue interaction [1]. The mechanism of material removal changes fundamentally with this parameter.

With longer pulses (typically nanoseconds or longer), the interaction is primarily photothermal. The absorbed energy dissipates as heat, leading to melting, explosive boiling, and material ejection. However, this process often causes undesirable side effects, including significant thermal damage to adjacent tissues, the formation of micro-cracks, and a layer of carbonization, all of which can compromise the quality of a dental restoration.

Conversely, ultrashort pulses from femtosecond (fs) lasers—whose duration is shorter than the time required for heat to diffuse into the surrounding material—confine the energy spatially and temporally. This enables non-thermal or "athermal" ablation mechanisms, such as plasma-mediated ablation, which vaporizes the material instantly while preserving the structural integrity of the adjacent tissue.

This fundamental advantage—the near-absence of collateral thermal damage—makes fs lasers an exceptionally promising tool for creating clean, precise, and highly reproducible cavities. Such high-quality preparations are essential for enhancing the bond strength of restorative materials and ensuring the long-term success of dental treatments. Consequently, a significant body of recent research has focused on identifying the optimal processing parameters, including fluence, pulse repetition rate, and scanning strategy, to maximize the efficiency and quality of fs laser ablation in enamel and dentin.

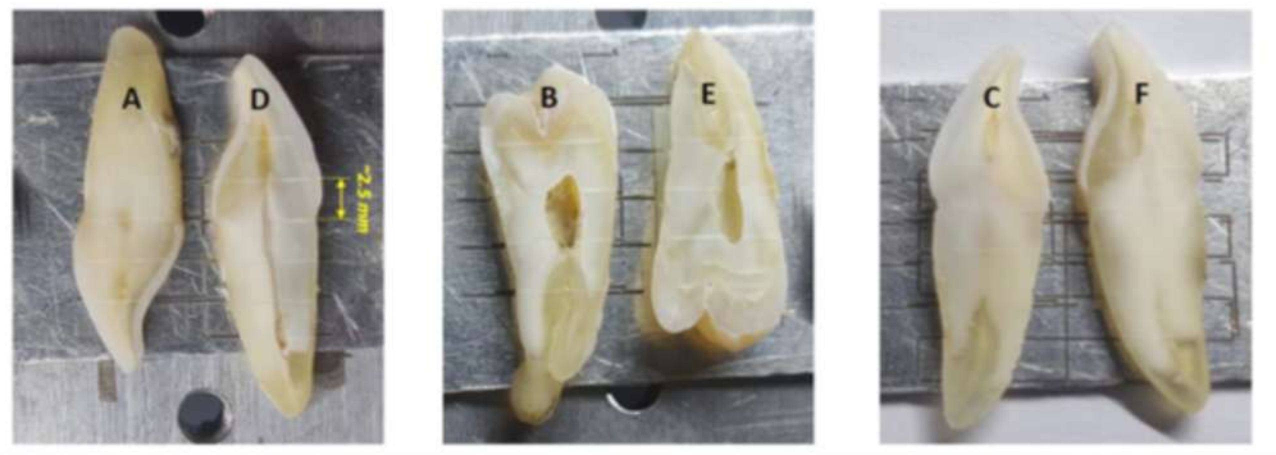
When fs laser pulses are applied, a high peak power is delivered to the samples in a very short timeframe, leading to negligible heat diffusion within the materials. Accurately describing the fs laser ablation process poses challenges due to the simultaneous occurrence of many physical phenomena. With fs lasers, the mechanism involves the ionization of atoms or molecules on the surface of the irradiated material through non-linear processes, resulting in the formation of a dense plasma. This plasma expands rapidly upon completion of the pulse (which lasts around 100 femtoseconds) without allowing heat to diffuse into the solid material. In this scenario, two competing mechanisms govern the ablation process: Coulomb explosion and thermal vaporization [2]. The Coulomb explosion becomes more significant when the laser intensity approaches the ablation threshold. Beyond this threshold, once the Coulomb explosion has occurred, thermal vaporization becomes the primary mechanism for material removal.

To characterize the changes induced in hard dental tissues by ablation processes, various techniques have been employed. Common methods include optical microscopy, scanning electron microscopy, X-ray diffraction, X-ray photoelectron spectroscopy, energy dispersive X-ray spectroscopy, 3D line scanning, and optical coherence tomography. Alternatively, researchers have utilized vibrational techniques, such as Fourier-transform infrared (FT-IR) and Raman spectroscopies, to assess the changes occurring in dentin and enamel tissues as a result of fs laser pulses [3–9]. These vibrational methods offer significant advantages for characterizing dental components and their potential biochemical changes.

In the present study, FT-IR spectroscopy was used to characterize the changes induced by fs laser ablation in human teeth extracted for orthodontic and periodontal reasons. The contributions of various dental components were identified, and comparisons were made with previous results. The analysis of vibrational spectra can help determine the optimal operating parameters for efficient laser ablation processes.

2. Materials & Methods

The human teeth were disinfected in 5% Sodium Hypochlorite solution (Ogna Lab, Florence Italy) for 24 h, dental pulp remanent was removed, and few-millimeter-thick slices were cut from human molar teeth using a diamond saw (Buehler, Lake Bluff, IL, USA). The samples were dried and rehydrated with distilled water for 24 h to restore the normal fully hydrated state. The laser ablation was performed in ambient air by using a chirped pulse amplification Yb: KGW laser source emitting pulses at a wavelength of 1030 nm, with a pulse duration of approximately 180 fs, and a maximum repetition rate of 200 kHz. Ablation rate of 1 kHz and peak fluences (Fp) the range of 0.7–1.5 J/cm^2 to generate regular line patterns on various samples.



$F_p \approx 1.5$ J/cm^2 $F_p \approx 1.0$ J/cm^2 $F_p \approx 0.7$ J/cm^2

Figure 1. Photograph of the tooth slices processed with 1030 nm fs pulses at three different fluences values F_P as indicated in the respective panels.

To analyze the processed tooth disks, micro-ATR (Attenuated Total Reflection) spectra were collected using a Perkin Elmer Spectrum One spectrometer equipped with a Mercury Cadmium Telluride detector (MCT) and a 0.6 mm radius germanium hemispherical internal reflection element (IRE). Using this configuration spectra were acquired in the ATR mode and the signal coming from a few μm-thick sample layer was collected. To obtain good quality spectra a contact between IRE and sample surface was necessary and particular care was employed in order to achieve the same degree of contact for all measurements. When contact was achieved, the ATR FT-IR spectrum of the selected area on the surface was collected. The germanium IRE allowed to acquire spectra from areas of the sample of 100 μm of diameter that can be properly chosen on the sample surfaces. The background spectrum was collected through the IRE when it was not in contact with the sample [10].

The spectra were acquired using 64 scans in the 4000 to 650 cm^{-1} with a 4 cm^{-1} spectral resolution facilitating the investigation of the chemical changes induced by the laser. High-quality spectra were acquired from treated and untreated regions of different samples.

3. Results and Discussion

FT-IR spectra of both untreated and treated teeth at various laser fluences are illustrated in Figures 2–4. Table 1 lists the positions of the main contributions along with their respective assignments in agreement with Refs. [3–6].

Table 1. Main peaks in FT-IR spectra of tooth samples (HA = hydroxyapatite; n = stretching vibration; d = bending vibration) according to Refs. [3–6].

Peak Position (cm^{-1})	Assignments
3265–3250	n OH^- n N-H (Amide A) of collagen
2920–2850	n C-H of organic compounds and contaminants

~1730	n C=O of carbonyl group
1628–1625	n C═O (Amide I) of collagen
1533–1520	n C—N and d N—H (Amide II) of collagen
1430–1422	n_3 $CO3^{-2}$ substituted in B-type PO^{3-}_4
~1393	n_3 $CO3^{-2}$
1231–1225	Amide III of collagen
~1003	n_1 PO^{3-}_4 of HA
947–941	n_1 PO^{3-}_4 of HA

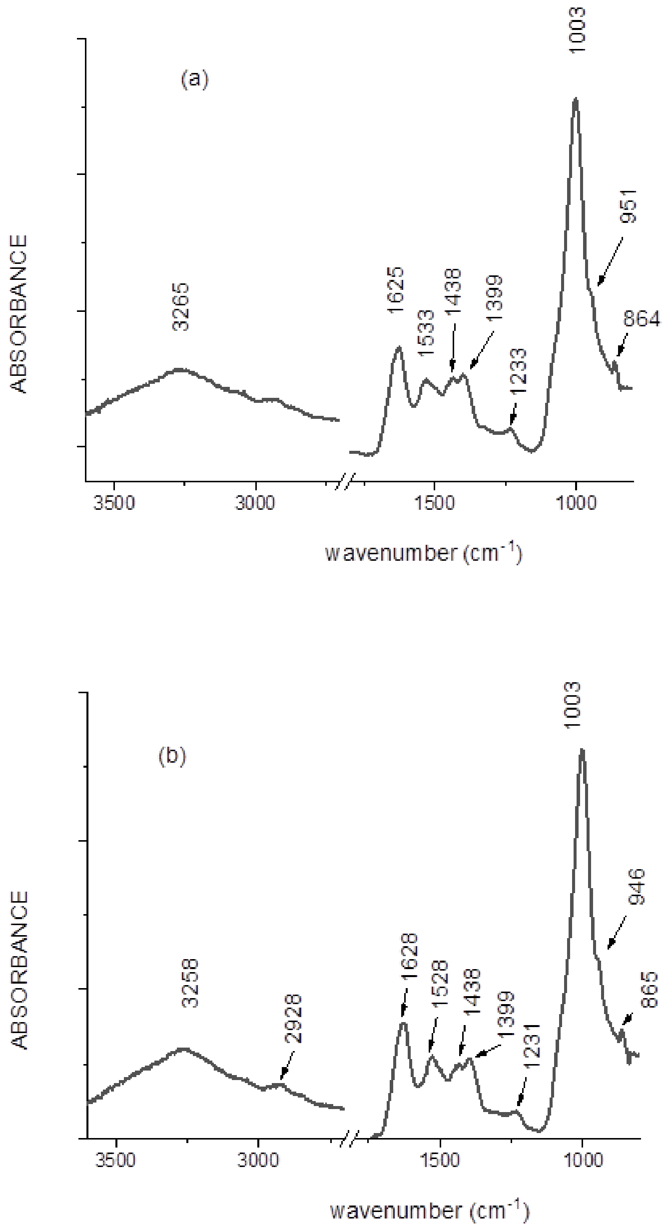


Figure 2. Representative spectra acquired from a (**a**) not treated region and (**b**) treated of sample C irradiated with a fluence of ≈0.75 J/cm^2.

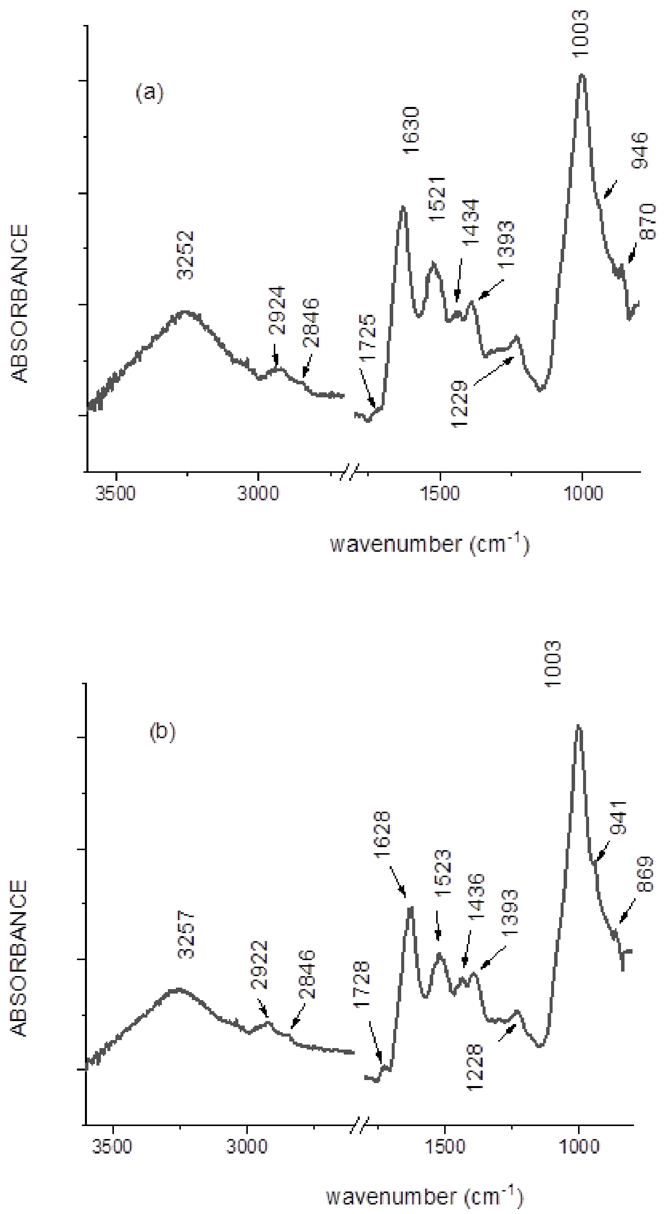


Figure 3. Representative spectra acquired from a (**a**) not treated region and (**b**) treated of sample B irradiated with a fluence of ≈1 J/cm².

The contributions of different dental components can be observed in the various spectra, showing small changes in intensity and position. From the spectra of untreated and treated regions presented in Figures 2 and 3, it is evident that there are no significant changes in the spectral features after laser treatments with fluence rates of 0.75 and 1.0 J/cm². Only minor adjustments in the peak positions have been noted. Specifically, the small changes in the region of 2920–2850 cm⁻¹, related to C-H contributions, have been attributed to laser treatment by previous authors [3,4], consistent with XRD (X-ray diffraction) and XPS (X-ray photoelectron spectroscopy) measurements.

For sample A, which was treated with a fluence of 1.5 J/cm², the situation is markedly different, as it was not possible to obtain high-quality spectra from the treated regions. This is likely due to significant morphological changes on the sample surface caused by the higher fluence rate. Although using an IRE (internal reflection element) for spectrum acquisition allows for mapping of the sample surface, good contact between the IRE elements and the sample surface is essential.

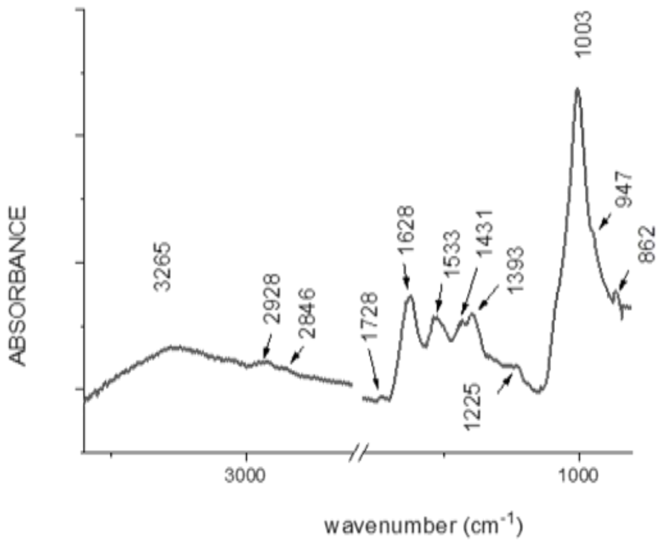


Figure 4. Representative spectra acquired from a not treated region of sample A irradiated with a fluence of ≈1.5 J/cm².

Despite this limitation, this investigation suggests that fluence rates between 0.75 and 1 J/cm² are suitable for laser ablation processes. To explore the effects of higher fluence rates, it is advisable to use spectroscopic techniques that do not require contact with the sample surface. Consequently, samples D, E, and F are currently being examined using Raman spectroscopy, which will also facilitate the acquisition of spectra from very small regions.

4. Conclusions

The reported results confirm that FT-IR spectroscopy in micro-ATR geometry enables the acquisition of spectra with good signal-to-noise ratios (SNR). These spectra can provide valuable information about the biochemical components of dental tissues and any changes that occur following fs laser treatments. When fluence rates ranging from 0.75 to 1 J/cm² are applied, no particularly noticeable changes have been observed. Further analysis of the small modifications in pulse shape is currently underway to fully utilize the capabilities of FT-IR spectroscopy.

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