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Original Article

Study Optical and Structural Properties of Polyaniline films using Laser Irradiation

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ABSTRACT

In this study, a thin film of poly(aniline) was prepared by spin coating. The aim of this study was to evaluate the effect of pulsed Nd: YAG laser irradiation on the properties of poly(aniline) thin films at a wavelength of 532 nm, a repetition rate of 5 Hz, and pulse energies of of 0,150, 250, and 350 mJ for a fixed duration of 30 seconds. The optical, structural, and surface properties of the films were analyzed before and after irradiation using UV–Vis, AFM, FTIR, and XRD.The results showed a decrease in the energy gap from 2.35 eV to 2.2 eV, an increase in the absorption coefficient from 0.025 cm^{-1} before irradiation to 0.033 cm^{-1} after irradiation, improved polymer chain regularity, and increased chain length and charge carrier mobility. An increase in surface roughness and improvement in microstructure was also recorded, without significant thermal damage, and an increase in particle size from 26.0 nm before irradiation to 363.5 nm after irradiation. The results also indicate that pulsed laser at 532 nm provides measurable and controllable modification of the optical, structural, and surface properties of poly(aniline).

Keywords: Poly(Aniline) (PANI), Pulsed Laser (Nd:YAG), Energy Gap, Optical Properties, Thin Films.

1 Introduction

Organic semiconductors, such as poly(aniline) (PANI), are promising materials in modern optoelectronic applications due to their low density, light weight, and high flexibility compared to inorganic semiconductors, as well as their ease of fabrication and low production cost [1,2]. Polyaniline has distinctive electrical and optical properties, such as good electrical conductivity, clear redox properties, and high thermal stability, making it suitable for a wide range of electronic and electrochemical applications [3,4]. It can also be easily prepared using chemical or electrochemical methods, even in a variety of organic solvents, allowing for precise control of the properties of thin films fabricated from it [5]. However, controlling the optical and electrical properties of these materials remains a major challenge, including energy gap, absorption coefficient, and charge transport, which directly affects the efficiency of optoelectronic devices such as OLEDs and organic photovoltaic (OPV) cells [6,7]. Therefore, understanding and developing methods to modify the optical and structural properties of organic polymers is essential to improve their performance in advanced optoelectronic applications.This study aims to explore the effect of pulsed laser processing on the optical and structural properties of thin poly(aniline) films, with a focus on improving absorption, reducing the energy gap, and increasing crystallinity, enabling the design of organic materials with adaptable properties to meet the requirements of future optoelectronic applications [8,9]. Some studies have addressed the effect of pulsed laser treatment on polyethylene membranes. A pulsed Nd: YAG laser with a wavelength of 532 nm was used, with a pulse duration ranging between 7–10 ns, a pulse energy between 50–200 mJ, and energy density (fluence) within the range of 0.5–2.5 J/cm², and the number of pulses ranged between 10–100 pulses. The structural, optical, and electrical properties were evaluated using FTIR, UV-Vis, and SEM techniques, in addition to measuring conductivity using the four-point probe method. The results showed a significant improvement in electrical conductivity at an energy density of 1.5 J/cm², due to the rearrangement of polymer chains, improved doping, and increased structural regularity.Compared to that study, the current study focused on analyzing the effect of pulsed laser treatment under specific operating conditions on the energy gap, absorption coefficient, and crystal structure of poly(aniline) films, with the aim of understanding the relationship between laser parameters and structural and optical changes [10].

2 Materials Used

A solution was prepared using (450mg) of poly(aniline) (PANI) basic emeraldine (Indian origin) with the molecular formula

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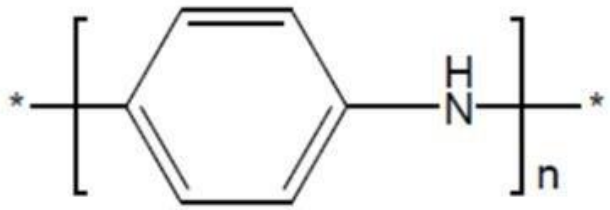

Figure 1: Chemical Structure of (Polyaniline)(PANI).

[C6H4NH]n and a purity of 98%, by dissolving it in 4 ml of chloroform, resulting in a final solution concentration of 112.5 mg/ml. The solution was mixed in a small flask to protect it from light rays, placed in a magnetic stirrer, and placed on a heater manufactured by FAITHFUL China to ensure the homogeneity of the mixture for 120 minutes. The homogeneous mixture was then exposed to pulsed laser radiation (Nd:YAG) produced by Beijin Sincoheren S&T Co.ltdav (China), with a spot size of 1 to 10 ml and a flux density of 10 to 2000 m J/cm-1At different energy levels of 0, 150, 250, and 350 mJ, for a fixed period of 30 seconds, with the samples placed at a distance of cm from the pulsed laser source 20 cm), at a fixed frequency (5 Hz), and the surrounding atmosphere is air at room temperature with a wavelength of 532 nm because it falls within the broad absorption band of polyaniline in the visible region (400–800 nm) associated with polaronic transitions improves chain regularity, increases conjugation length, and enhances charge carrier mobility, with noticeable changes in optical, structural, and surface properties. The solution was then deposited on glass slides with a thickness of (200 nm) using a spin coating device at a rotation speed of (3500) rpm for one minute to form thin films with a thickness of (0.5 µm). The optical properties of the samples were examined before and after exposure to pulsed laser using ultraviolet spectroscopy (UV–NIR) technology produced by Metertech SP-8001 (Taiwan). This was done to analyze the absorption spectrum, reflection, and permeability. The chemical composition of the produced thin films and their functional groups were determined by analyzing the molecular fingerprint of the materials involved in the chemical processes using Fourier transform infrared spectroscopy (FTIR) (JASCO, Japan). Structural changes were also evaluated using an atomic force microscope (AFM) (manufactured by Nanosurf, Switzerland, 2025) to study surface morphology and roughness. The crystalline and chemical structure was also determined using X-ray diffraction (XRD) (manufactured by Panalytical, Netherlands, 2024).

3 Results and Discussion

3.1 Optical Properties

3.1.1 Optical Absorption and Absorption Coefficient

Figure 2 shows the intensity of the absorption spectra for identifying the peaks to determine the composition of the thin poly(aniline) films before and after laser exposure. The membranes prepared before irradiation show an absorption intensity of 1.3, where the absorption spectrum of non-laser-exposed poly(aniline) (PANI) shows clear peaks in the range of ≈ 400–450 nm (π→π* range) associated with electronic transitions linked to the conjugated network, reflecting an extension in the electronic coupling system that determines the energy gap and electronic structure of poly(aniline) [11]. After exposing the membranes to a pulsed laser beam with an energy of 150 mJ, the absorption intensity increases to 1.4. In other words, exposure to Nd: YAG pulsed laser beams alter the nonlinear optical properties of polymer systems (such as PANI and its composites), for example, changing the absorption of materials and the extent of interaction with light energy according to the intensity of the laser energy. With an increase in laser energy (250 mJ and 350 mJ), we observe a gradual decrease in the intensity of the absorption peaks in the UV-Vis spectrum, where the absorption intensity reached (1.05) at an energy of (350 mJ) and reached (1.0) at an energy of (250 mJ), as a result of the effect of high temperature and structural defects or due to the breakdown of the electronic coupling system on the polymer network, leading to an increase in the energy gap and thus reducing absorption in certain visible ranges [12].

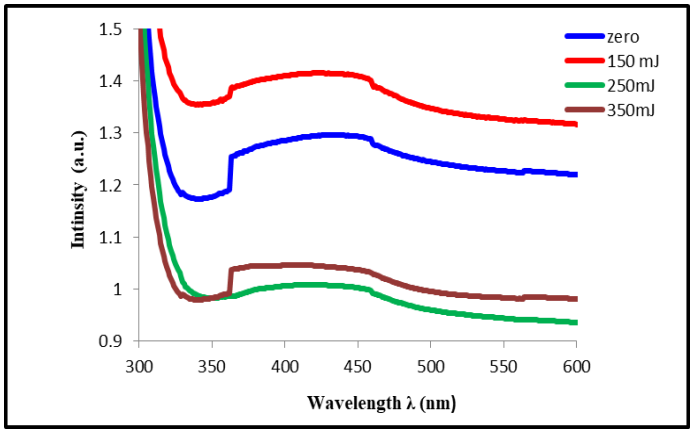

Figure 2: Absorption intensity spectra of thin poly(aniline) films before and after exposure to different laser powers.

The study focuses primarily on the maximum absorption value observed at an irradiation energy of 150 mJ, which represented the optimal energy for improving the photoabsorption of thin films. The absorption results shown in Figure 3 for thin poly(aniline) membranes before laser exposure showed an absorption of approximately 0.013, and after exposure to laser pulses with an energy of 150 mJ, it reached 0.014 at a wavelength of approximately 450 nm. Exposure to laser pulses led to a significant increase in absorption intensity as a result of breaking the bonds directly within the polymer chains, which contributed to enhancing the optical and structural properties of the material [13].

The optical absorption coefficients used in the Tauc equation to calculate the energy gap were determined using equation (1) [14].

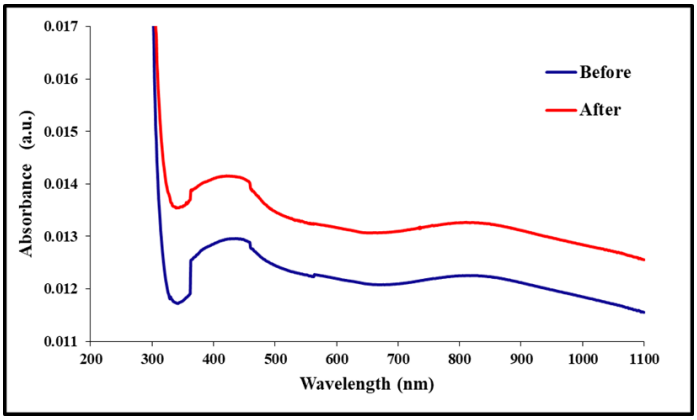

Figure 3: Absorption spectra of thin PANI films before and after exposure to pulsed laser radiation (150 mJ).

$$\alpha = \frac{1}{d} ln + \left[\frac{(1-R)^2}{2T} + \sqrt{\frac{(1-R)^4}{4T^2} + R^2} \right] \quad (1)$$

The variables α, d, T, and R represent the absorption coefficient, thin film thickness, permeability, and reflectance, respectively. Figure 4 shows the results of the absorption coefficient values for both the untreated polyethylene film and the film exposed to a 150 mJ pulsed laser. The results indicate that pulsed laser treatment led to a noticeable increase in the absorption coefficient values across wavelengths nm(300-1100), with a post-treatment value of approximately (0.033 cm-1) compared to a pre-laser exposure value of approximately (0.025 cm-1). This increase indicates a change in the electronic structure of the film. The pulsed laser energy caused strong electronic excitation within the polymer chains, thereby increasing the probability of ππ* transitions and polarone transitions within the energy range. This type of change is associated with an increase in the density of allowed electronic states, leading to an increase in the total absorption coefficient of the film [15]. These results are consistent with recent studies confirming that laser treatment is an effective technique for controlling the optical and nonlinear properties of conductive polymers [16].

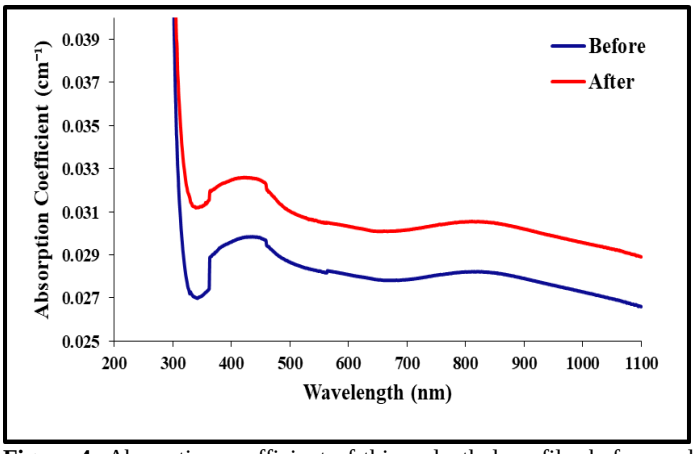

Figure 4: Absorption coefficient of thin polyethylene film before and after exposure to pulsed laser radiation with a power of 150 mJ.

3.1.2 Transmittance and Reflectivity Spectra

Figure (a-5) shows a significant decrease in transmittance after irradiating the poly(aniline) (PANI) film with a pulsed laser (150 mJ) compared to the pre-treatment condition, where its value before exposure to the laser was (0.971). and after irradiation, it was approximately 0.969. This decrease indicates an increase in the optical absorption coefficient, which reflects a change in the electronic structure of the polymer. Absorption in the near-ultraviolet region ≈ nm (300–350) is due to ππ transitions within the aromatic rings of the polymer chain, while in the visible and near-infrared regions, absorption is associated with polarone and bipolarone transitions[17]. Figure (b-5) also shows an increase in reflectivity after irradiation of approximately (0.018) compared to the pre-irradiation state, where its value was (0.016). According to Frenel's equations, an increase in absorptivity and conductivity leads to an increase in surface reflectivity[18]. The improvement in reflectivity in this spectral range is the result of molecular rearrangement, in addition to an increase in material ensity caused by changes in chemical bonds and structural rearrangement under the influence of pulsed laser [19].

3.1.3 Refractive Index

Figure 6 shows the refractive index (n) of poly(aniline) (PANI) films as a function of wavelength before and after irradiation with a pulsed laser at 150 mJ. The results show a clear increase in the refractive index after laser treatment, due to an increase in the absorption coefficient after treatment. We note that the refractive index increased after irradiation to a value of approximately 1.31, compared to 1.29 before irradiation within the wavelength range of 400-450 nm[20].

Calculating the refractive index using equation (2) is essential for studying optical properties in materials science.

$$n = \left(\frac{1+R}{1-R}\right) + \sqrt{\frac{4R}{(1-R)^2} - K^2} \quad (2)$$

This change is also caused by the polarization of carbon-carbon (C–C) bonds within the polymer chains. As a result of these effects, reactions occur at the surface interface, which stimulate photochemical reactions on the surface of the poly(aniline) material and contribute to the rearrangement of the polymer chains. As a result, the structural and optical properties of the material are improved.

3.1.4 Optical Energy Gap (Eg) of Poly(aniline)Films

The energy gap is one of the fundamental criteria that distinguishes semiconductors from insulators, given its prominent importance in the design and development of these organic materials. This gap can be used to determine the electrical and optical properties of materials, making it very important for applications that require control of electrical conductivity and the interaction of the material with light [7]. To extract the energy gap value, we use the Tauc equation, which expresses the relationship between absorptivity and photon energy as follows:

$$(ahv)^n = K(hv - Eg) \quad (3)$$

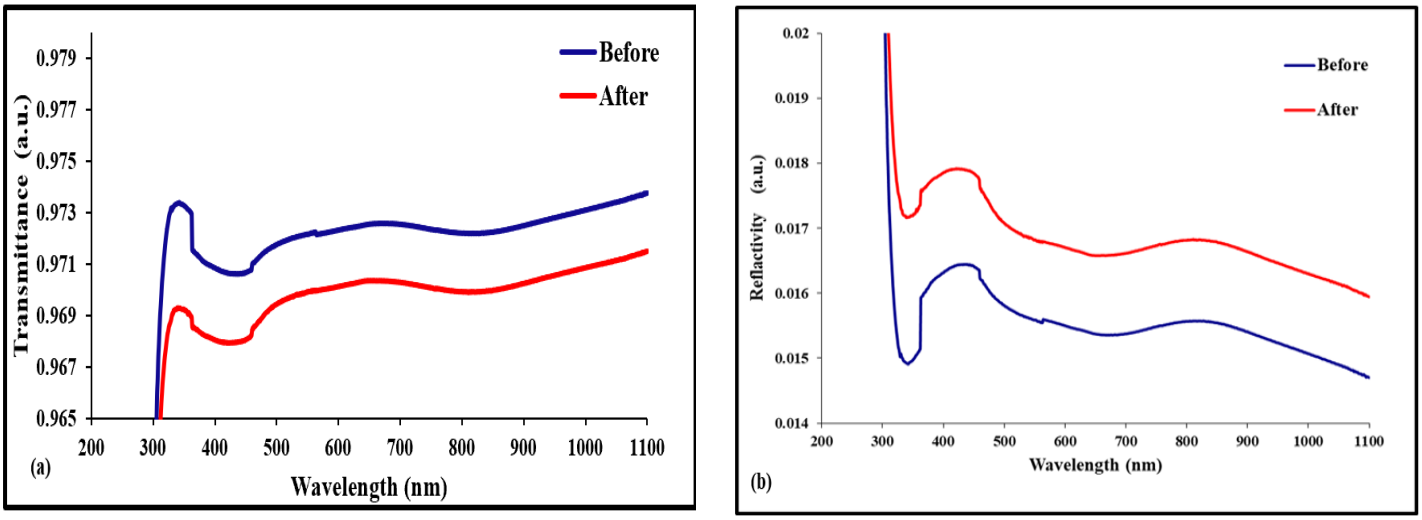

Figure 5: (a) Light transmittance before and after irradiation of the poly(aniline) film with a pulsed laser (150m J) (b) Reflectance of the poly(aniline) film before and after irradiation with a pulsed laser (150m J).

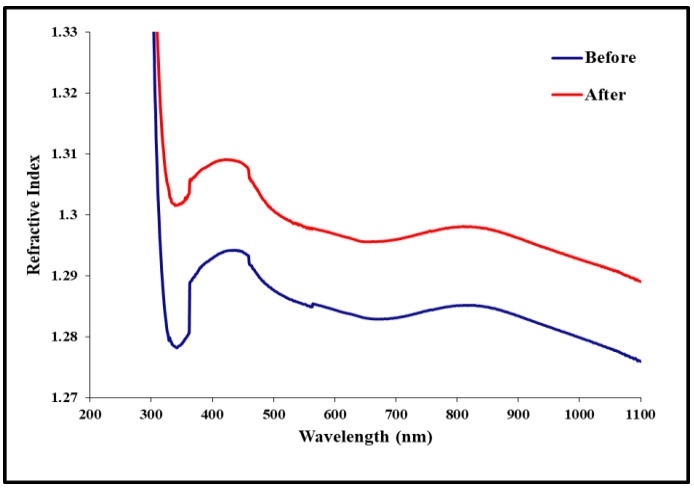

Figure 6: Refractive index of poly(aniline) film before and after pulsed laser treatment Energy(150mJ).

Where:

hv: represents the photon energy, a: absorption coefficient, Eg: band gap energy K: constant depending on the type of material, n expresses the type of electronic transition (direct or indirect, allowed or forbidden). In the first spectrum, the optical absorption curve (blue line) of the poly(aniline) film before laser treatment shows a gradual increase in absorption with increasing photon energy, indicating that the material begins to absorb light. Figure 7 shows the optical energy gap (Eg) of poly(aniline) films, where the y-axis represents (ahv)2 and the x-axis represents photon energy hv. Direct transitions are allowed because the electronic structure of PANI supports this type of transition in this energy range [21,22]. The optical energy gap before laser exposure was determined to be approximately 2.35 eV [7,23]. After laser treatment (red line in the spectrum), a significant decrease in the energy gap value to approximately 2.2 eV was observed, indicating a change in the molecular structure of the material as a result of laser irradiation, which led to a reduction in the optical energy gap and facilitated electronic transitions [8,9].The decrease in the energy gap is due to improved molecular coherence and the introduction of new local energy levels (polaron or dipolaron) within the gap, strongly suggesting that laser treatment significantly affects the structural composition of poly(p-phenylenediamine). By facilitating electronic transitions, the new energy levels contribute to the improvement of the optoelectronic performance of poly(p-phenylenediamine).As a result of these optical changes, the material performs better in light harvesting and sensing devices, including photodetectors, optical sensors, and solar cells [24,25].. Energy gap values are a key metric when selecting organic semiconductors for various applications, as they significantly affect the electrical and optical properties of materials [26]. Laser processing can also break the crystal lattice and internal structural defects, which can lead to the overlap of energy levels between the HOMO and LUMO orbitals, thereby reducing the bandgap [27].

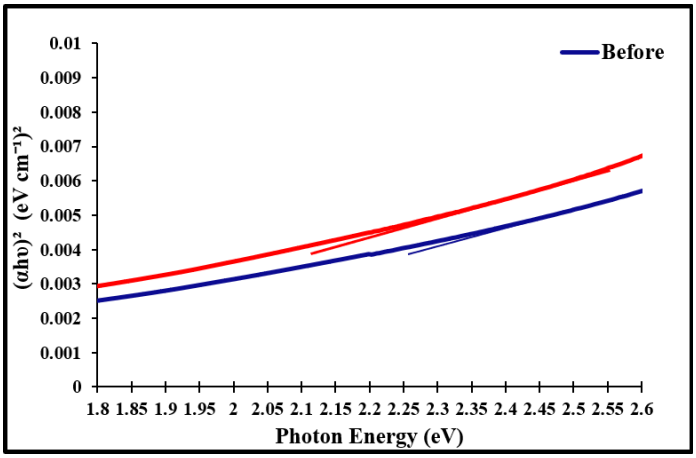

Figure 7: Energy gap of thin poly(aniline) films before and after irradiation with a pulsed laser (150 mJ).

3.2 Fourier transform infrared spectroscopy (FTIR)

The thin poly(aniline) films were chemically studied and the effect of laser irradiation on their molecular structure was examined using Fourier transform infrared spectroscopy [28].

Figure 8 shows the results of comparing the infrared transmittance spectra before and after treating the poly(aniline) thin films with a pulsed laser with an energy of 150 mJ. Using this technique, we can determine the chemical bonds in the material and the extent of the effect of the treatment on the molecular structure of poly(aniline) [29]. The FTIR spectra of poly(aniline) before laser irradiation in Figure (8-a) show several peaks, including one at 700 cm^{-1} and another at 900 cm^{-1}. These peaks indicate a C–H bond representing out-of-plane vibrations on the aromatic benzene ring of the polymer [30]. The peak at 1500 cm^{-1} indicates stretching vibrations in the C–N bond, indicating the presence of a polymer structure containing these bonds [31]. The range between 1600 and 1500 cm^{-1} shows that poly(aniline) has an aromatic structure, as the peaks in this range indicate vibrations of double C=C bonds in the aromatic rings [32]. Finally, the peaks at 2123–3000 cm^{-1} represent stretching vibrations in the C≡C bonds in the alkyne groups and C≡N bonds in the nitrile groups. After laser treatment of poly(aniline), the following spectra were recorded: The decrease in absorption in the range between 1000–400 cm^{-1} shows the effect of the laser on the chemical bonds, resulting in changes in the molecular structure [33]. New peaks appeared in the range between 4000–400 cm^{-1}, indicating changes in the molecular structure of the material as a result of laser treatment. A decrease in transmittance (%T) was also observed at certain frequencies after treatment, indicating an increase in light absorption from the material. This is evidence of a change in optical properties [34]. By comparing the spectra before and after laser treatment, it is clear that the treatment significantly affects the molecular bonds in poly(aniline), leading to changes in the chemical structure, such as the appearance or disappearance of certain peaks and shifts in the intensity of vibrations. These changes indicate an improvement or modification in the mechanical, optical, and electrical properties of poly(aniline), as shown in some recent studies [3,35]. The main chemical bonds after treatment with a pulsed laser (150mJ) in the material are shown in Figure (b-8) (peaks at 600, 700, 900, 1000, 1100, 1500, 1700, 1800, 2800, 2900, 3100, 3300, and 3600) cm^{-1}, where these peaks represent, respectively: C–H vibrations, =C–H bending, C–N stretching, C–C stretching in the ring, –C=C– stretching, C–H stretching, =C–H stretching, one group, and –C≡C–H stretching.

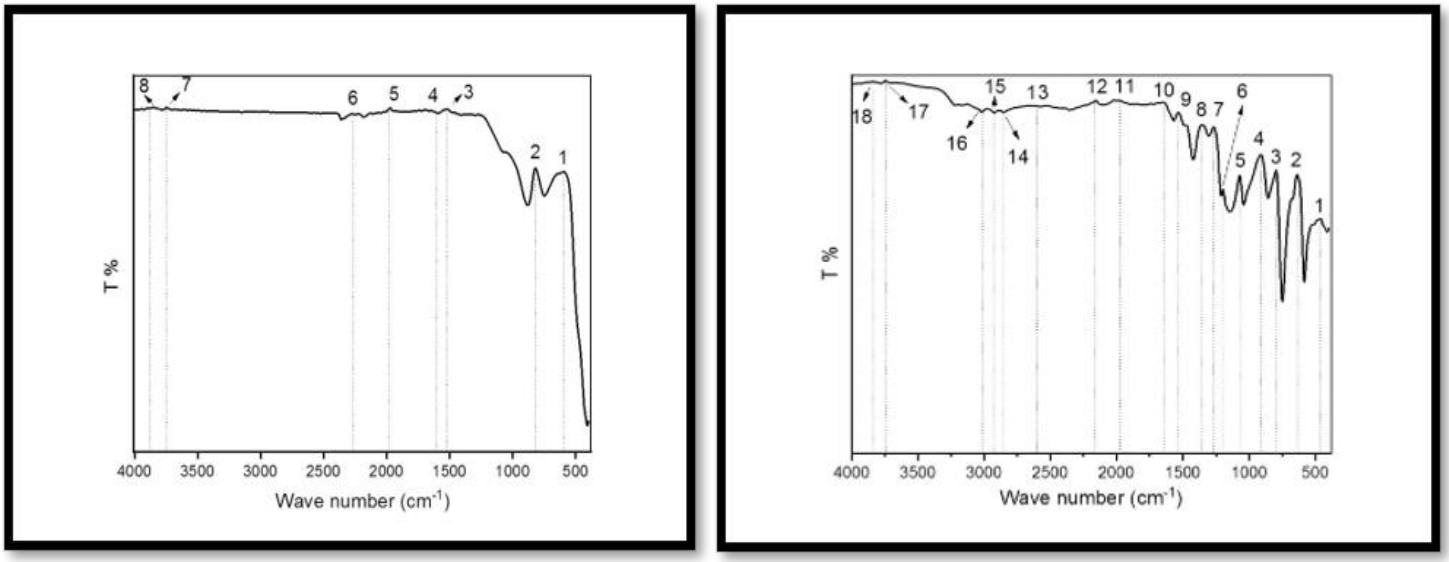

Figure 8: Infrared transmission spectra (FTIR) of thin poly(aniline) films (a) before laser treatment. (b) After exposure to pulsed laser radiation with an energy of 150 mJ.

3.3 Structural Properties

3.3.1 X-Ray Diffraction (XRD)

X-ray diffraction is the primary technique for determining the crystalline and chemical structure of poly(aniline), including the distance between crystal planes (d-spacing), crystal size, lattice constants, and full width at half maximum (FWHM) [36]. By analyzing thin poly(aniline) films, it is possible to reveal their chemical and structural composition and other properties. This data is essential for studying the structural behavior of the material and evaluating the molecular interactions between neighboring molecules [18]. These secondary chemical interactions significantly affect the electrical behavior of the material [19]. Figure 9 shows the XRD spectrum of the thin poly(aniline) film before irradiation, where the horizontal axis represents the diffraction angle (2θ) in the range (10-90) and the vertical axis represents the diffraction intensity in the range (100-400-900). The results before laser irradiation showed diffraction peaks at angles (θ = 9.0837°) and (θ = 15.5927°).

Using the peak width at half height for the high-intensity peaks, the crystallite size can be calculated using the Scherrer equation shown in equation (4) below:

$$\frac{K\lambda}{\beta \cos\theta} \quad (4)$$

Where:

D represents the crystal size, K is the shape factor (0.9), λ is the X-ray wavelength (nm0.15406), β is the full width at half maximum (FWHM), θ is the Bragg angle, and the data used to determine the crystal size is shown in Table (1). Figure (10)

shows that the X-ray diffraction spectrum exhibited new diffraction peaks, as shown in the data in Table (2). The appearance of these peaks is attributed to spatial changes in density within the deposited polymer volume [20]. Laser treatment also showed an increase in the angle at which X-ray diffraction peaks appear. This change leads to compression of the crystal units and a decrease in lattice constants, indicating a phase transition [21]. In addition, the value of the distance between crystal planes (d-spacing) corresponding to the angle (90.0837) before laser treatment (9.72757 Å) decreased to (3.97522 Å) corresponding to the angle (220.3464) after pulsed laser treatment. Since the treatment process absorbs energy, it breaks the molecular bonds within the material, thereby fragmenting the molecules and removing part of the material with little thermal damage. This results in a change in the distance between the crystal planes and the size of the crystals. Furthermore, the material can be efficiently removed without significant thermal damage as a result of the dynamic interaction between the laser and the material, as the polymer chains are broken directly. This leads to noticeable changes in the size of the crystals and the distances between the crystal planes as a result of the incident energy [37].

The crystal size was determined using the Scherrer equation based on the diffraction parameters [23,24]. The crystal size was calculated before laser irradiation at the main peak located at the diffraction angle (2=90.0837), where the particle size was (26.0 nm), which is consistent with studies indicating that chemically prepared poly(aniline) has a semi-crystalline nanostructure [38].

After laser exposure, a clear increase to about (363.5 nm) was observed at the peak that appeared at the diffraction angle (2=220.3464). The increase is attributed to the pulsed laser energy, which stimulated crystal growth and reduced structural defects, thereby increasing crystallinity [39]. This is considered an improvement in the structural properties of thin polyamide films [40].

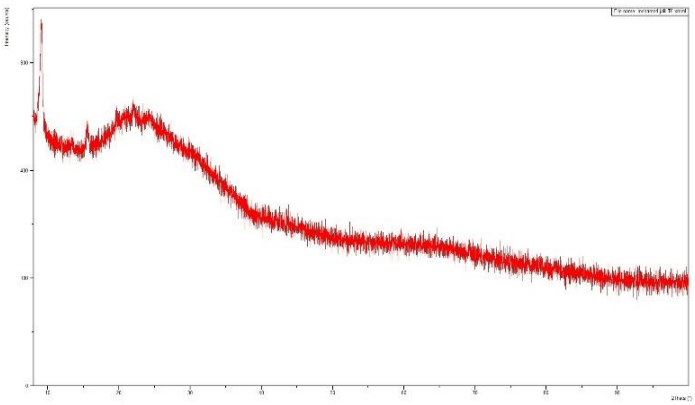

Figure 9: The Diffraction peaks for PANI film before pulse laser treatment.

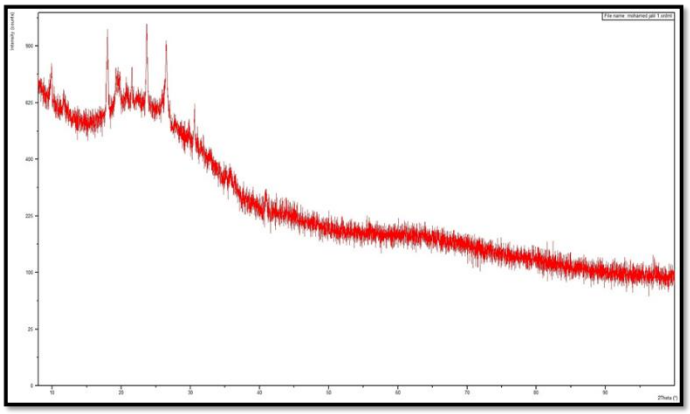

Figure10: The Diffraction peaks for PANI film after pulse laser treatment.

Table 1: XRD peaks and structural characteristics for PANI film before pulse laser treatment.

Pos. (2θ) °	Height [cts]	FWHM Left (2θ) °	d-spacing [Å]	Rel. Int. [%]
9.0837	340.38	0.3065	9.727157	100.00
15.5927	51.51	0.0753	5.67847	15.13
22.0577	136.98	3.0863	4.02659	40.24

Table 2: XRD peaks and structural characteristics for PANI film after pulse laser treatment.

Pos (2θ) °	Height [cts]	FWHM Left (2θ) °	d-spacing [Å]	Rel. Int. [%]
9.8445	80.20	0.2719	8.97744	6.07
11.7258	35.17	0.3956	7.54098	2.66
17.9654	231.75	0.4388	4.93349	17.55
19.2253	123.00	0.2127	4.61294	9.32
19.7212	145.29	0.6620	4.49806	11.00
20.7841	54.40	1.5854	4.27036	4.12
21.6959	138.10	1.9724	4.09290	10.46
22.3464	1320.29	0.0220	3.97522	100.00
23.7623	193.60	1.5702	3.74145	14.66
26.3871	282.16	1.2353	3.37493	21.37
28.9386	95.48	1.9357	3.08291	7.23
22.3464	1320.29	0.0220	3.97522	100.00
23.7623	193.60	1.5702	3.74145	14.66
26.3871	282.16	1.2353	3.37493	21.37
28.9386	95.48	1.9357	3.08291	7.23

3.3.2 Atomic Force Microscope (AFM)

Atomic force microscopy analyses were performed on thin poly(aniline) films before and after exposure to pulsed laser light with an energy of 150 mJ, in order to evaluate the surface morphology and roughness, as shown in Figure 11 in two- and three-dimensional configurations, respectively [41]. The results

showed that the root mean square (RMS) roughness of the thin film before irradiation was 10.699 nm, while the average surface roughness of these films was 6.038nm. After exposure to a pulsed laser with an energy of 150 mJ, the average root mean square roughness became approximately 89.095 nm, and the average surface roughness reached 60.02nm. The differences in grain size between the treated and untreated membranes indicate that the polymer structure underwent a transformation in crystallinity and self-organization. The significant increase in surface roughness observed in the AFM analysis after laser treatment is closely related to the XRD results. The increased peak intensity in the XRD patterns indicates a reorganization of the PANI polymer chains and an increase in crystallinity, which is physically manifested as a more defined surface topography and roughness. The decrease in permeability is directly proportional to the increase in RMS peaks, indicating that light scattering due to surface roughness is a major factor. The surface change upon exposure to high-energy pulsed laser light is evident from the increase in roughness. The structures also follow a typical polymer pattern, with well-defined parallel ripples within a small range of influence; this finding is consistent with the results of Ripolar [42].

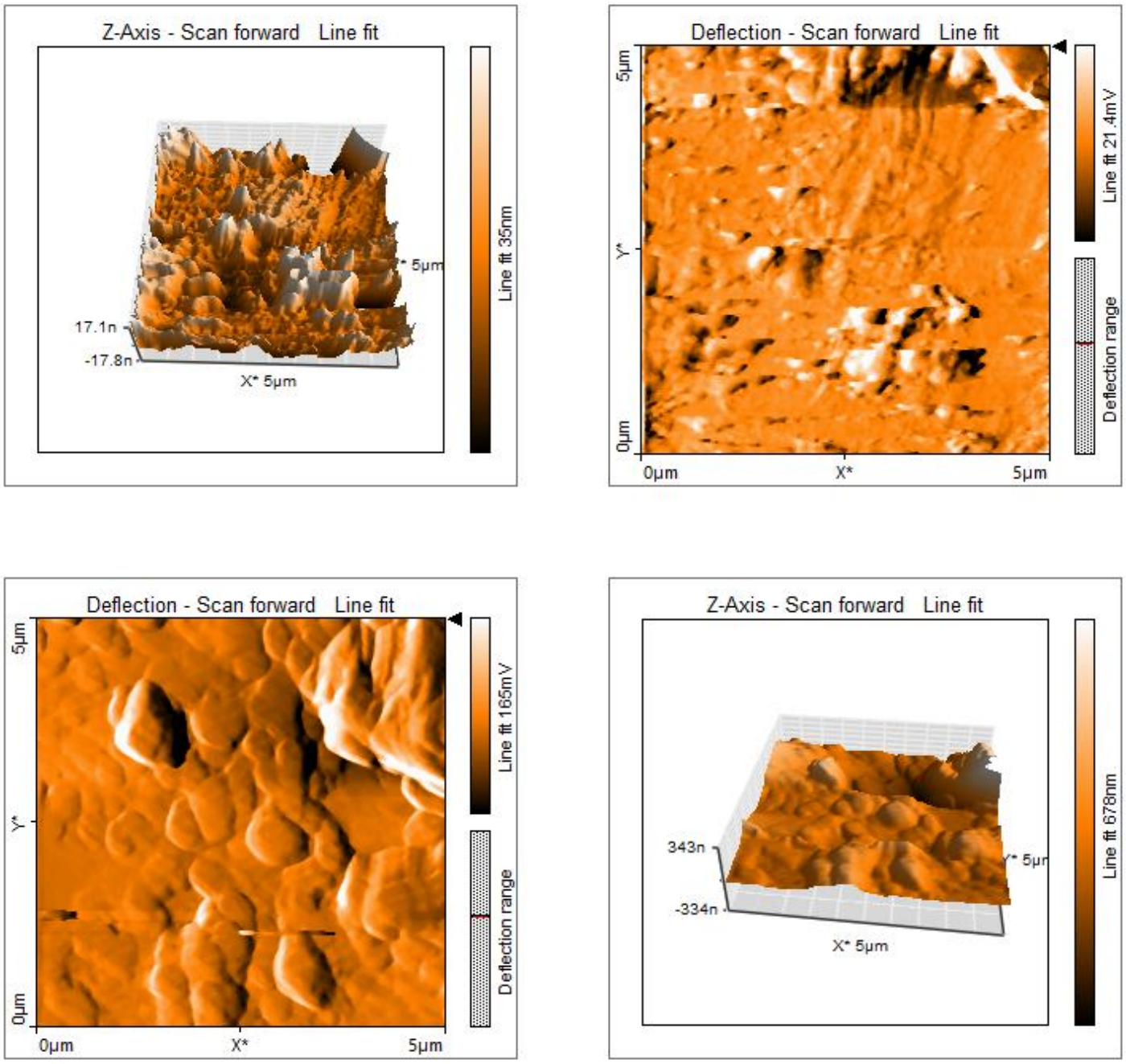

Figure 11: Two-dimensional and three-dimensional AFM images of thin poly(aniline) films (a) before laser irradiation (b) after irradiation with a pulsed laser (150 mJ).

Conclusions

This study examined the effect of pulsed laser treatment on the optical and structural properties of thin poly(aniline) films. The absorption spectra of the laser-treated samples showed a clear improvement as a result of the direct decomposition of the molecular chains within the polymer matrix, with the results showing an increase in the values of absorptivity, absorption coefficient, refractive index, and reflectivity, and a decrease in both the transmittance value and the optical energy gap. FTIR analyses also showed distinct peaks across the spectrum, resulting from the interaction of aromatic C–H vibrations with CH–C–CH, C–C, C–N, C=N, and N–H vibrations, indicating the preservation of important functional groups during laser treatment. XRD data after laser exposure showed peaks at higher angles, indicating a phase transition that led to compression of the crystal units and reduction of lattice constants. AFM results showed that poly(aniline) films experienced an increase in average surface roughness as well as root mean square (RMS) roughness, indicating an improvement in surface morphology and an increase in particle size. Overall, the results confirm that pulsed laser treatment is effective in modifying the optical and structural properties of thin poly(aniline) films, opening up new opportunities for tailoring their properties to meet the requirements of advanced optoelectronic applications.

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