



Structural, optical and electrical investigation of plasma-treated PANI nanostructures for solar cell application

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Polyaniline (PANI) has emerged as one of the most important conducting polymers in optoelectronic and photovoltaic (PV) devices because of its excellent environmental stability, high electrical conductivity, and tunable optical properties. For examining the impact of plasma processing on morphological, structural, optical, electrical, and PV properties of PANI nanostructures, they are created and altered utilizing plasma treatment for varying exposure times. A total of three samples has been analyzed: PANI treated with plasma for one minute, PANI as-prepared, and PANI treated with plasma for two minutes. Following plasma treatment, X-ray diffraction (XRD) investigation showed improved structural ordering and crystallinity, with distinctive diffraction peaks seen at roughly $2\theta = 20^\circ$, 25° , and 45° , that correspond to (020), (200), and (220) planes, respectively. FESEM and AFM analyses showed significant surface evolution, accompanied by an increase in average roughness (Ra) from 18.4 nm for the untreated sample to 24.9 nm and 31.6 nm for the 1 min and 2 min plasma-treated samples, respectively. FTIR spectra confirmed the preservation of the characteristic PANI structure with enhanced conjugation and molecular ordering after plasma exposure. UV-Visible analysis demonstrated improved optical transmittance and a gradual reduction in optical band gap from 3.18 eV (as-prepared) to 3.05 eV and 2.91 eV for the 1 min and 2 min treated samples, respectively. Electrical conductivity increased significantly from $1.82 \text{ S}\cdot\text{cm}^{-1}$ to $3.46 \text{ S}\cdot\text{cm}^{-1}$ and $5.71 \text{ S}\cdot\text{cm}^{-1}$ after 1 min and 2 min plasma treatment, respectively. Consequently, the photovoltaic performance improved substantially, with the power conversion efficiency increasing from 1.84% for the untreated film to 2.76% and 4.12% for the 1 min and 2 min plasma-treated samples, respectively. These findings demonstrate that plasma treatment is simple, cost-effective, and environmentally friendly approach for enhancing the performance regarding PANI nanostructures for solar energy conversion applications.

Keywords: Polyaniline; Plasma treatment; Optical; Electrical; Solar cells.

1. INTRODUCTION

The growing need to have sustainable sources of energy in the world has intensified the development of materials into highly advanced material to be utilized in photovoltaic applications. One of the major abundant as well as eco-friendly renewable energy sources is solar energy [1]. However, decreasing the production costs and improving solar cell performance are still crucial. Because of their low processing costs, tunable electrical characteristics, mechanical flexibility, and compatibility with wide area manufacturing techniques, conducting polymers emerged as attractive materials for next-generation PV devices [2]. One conducting polymer that has garnered a lot of attention is PANI, which combines electrical conductivity, chemical stability, environmental sustainability, and ease of synthesis. Doping, chemical treatment, and surface engineering are some of the ways to alter the electrical and optical characteristics of PANI [3].

These properties render PANI a good candidate in applications in sensors, supercapacitors, batteries, light-emitting devices and solar cells. Surface modification methods are commonly used so as to enhance the performance regarding nanostructures made of polymers. A very useful technique is the plasma treatment which could be utilized for modifying the surface characteristics of materials without compromising on their bulk properties. Plasma exposure is capable of adding new functional groups, enhancing surface energy, altering roughness and enhancing charge transport properties [4]. The interaction of the active layer with incident light can be dramatically affected by these changes and efficiency of carrier collection in photovoltaic devices is increased. A number of studies have shown that the morphology and conductivity of conducting polymers can be enhanced using plasma processing [5]. In PANI nanostructures, plasma treatment may cause structural rearrangement, enhance the surface roughness, and form new pathways of charge transport. These alterations are likely to enhance the optical absorption and electrical conductivity that are important parameters in solar cell applications [6].

Plasma exposure on PANI nanostructures needs a detailed characterization procedure to comprehend its impact. X-ray diffraction (XRD) can give data on structural ordering and crystallinity. Morphological changes and surface roughness can be observed utilizing scanning electron microscopy (SEM) as well as atomic force microscopy (AFM). Furthermore, Fourier-transform infrared spectroscopy (FTIR) recognizes the changes in chemical bonding and functional groups. UV-Visible spectroscopy makes it possible to assess optical absorption and band gap energy, whereas electrical measurements can help to understand the process of charge transport. In the current work, PANI nanostructures are made and exposed to plasma treatment for exposure times of 1 min and 2 min. Systematic investigation and comparison of the morphological, electrical, optical, and structural properties regarding the films are carried out and compared with the untreated films. Moreover, the applicability related to plasma-treated PANI films in solar cells is considered. The purpose of the study is to find out the correlation between plasma treatment duration and the material properties that are obtained, which can be of great significance to optimize the conduction of the polymer based photovoltaic devices.

2. MATERIALS AND METHODS

2.1. Materials

Aniline monomer is (C_6H_7N , purity $\geq 99.5\%$, molecular weight: $93.13 \text{ g}\cdot\text{mol}^{-1}$) has been obtained from Sigma-Aldrich, USA. Hydrochloric acid (molecular weight: $36.46 \text{ g}\cdot\text{mol}^{-1}$, HCl, ACS reagent grade, purity 37 wt.% in water) purchased at Sigma-Aldrich, USA. Ammonium persulfate (APS, $(NH_4)_2S_2O_8$,

purity $\geq 98\%$, molecular weight: $228.20 \text{ g}\cdot\text{mol}^{-1}$) is supplied by Sigma-Aldrich, USA. Acetone ($\text{C}_3\text{H}_6\text{O}$, purity $\geq 99.5\%$, molecular weight: $58.08 \text{ g}\cdot\text{mol}^{-1}$), ethanol (molecular weight: $46.07 \text{ g}\cdot\text{mol}^{-1}$, $\text{C}_2\text{H}_6\text{O}$, purity $\geq 99.8\%$), and deionized water are used as cleaning solvents for substrate preparation. All chemicals have been of analytical grade and utilized with no further purification. Microscope glass substrates ($25 \text{ mm} \times 25 \text{ mm} \times 1 \text{ mm}$) have been ultrasonically cleaned in ethanol, acetone, and deionized water for 15 min each, followed by drying under ambient conditions prior to thin-film deposition. Table 1 presents the specifications of the chemicals employed in this study, including molecular weight, purity percentage, supplier information, and country of manufacture.

Table 1 Chemicals that used in this study, the supplier is Sigma-Aldrich from USA.

Chemical	Molecular Formula	Molecular Weight ($\text{g}\cdot\text{mol}^{-1}$)	Purity (%)
Aniline	$\text{C}_6\text{H}_7\text{N}$	93.13	≥ 99.5
Hydrochloric acid	HCl	36.46	37 wt. %
Ammonium persulfate	$(\text{NH}_4)_2\text{S}_2\text{O}_8$	228.20	≥ 98
Acetone	$\text{C}_3\text{H}_6\text{O}$	58.08	≥ 99.5
Ethanol	$\text{C}_2\text{H}_6\text{O}$	46.07	≥ 99.8
Deionized water	H_2O	18.02	≥ 99.9

2.2 PANI nanostructures preparation

The PANI nanostructures are prepared through chemical oxidative polymerization and subsequently spin-coated onto glass substrates. First, 0.1 M aniline monomer has been dissolved in 1.0 M HCl solution, while the mixture has been continuously stirred for 30 minutes at room temperature for creating homogenous precursor solution. The oxidizing agent is 0.1 M ammonium persulfate (APS) dissolved in 1.0 M HCl in different breaker. Under continuous magnetic stirring, the APS solution has been added dropwise to aniline solution. For complete polymerization, the molar ratio of aniline to APS (1:1). For controlling the reaction rate and produce homogeneous polymer chains, the polymerization procedure is conducted in ice bath (0-5) °C.

Constant stirring has been continued up to 6 h where the solution would slowly turn from colorless to dark green, which indicates emeraldine salt formation, the conductive form of PANI. Once the reaction has been complete, the resulting polymer precipitate is left to age 12 hrs to ensure complete polymerization. The PANI which had been synthesized is filtered and washed repeatedly with deionized water as well as ethanol until filtrate became colorless and pH is neutral. Also, the purified polymer is dried in hot-air oven at a temperature of 60 Celsius for 12 hrs to eliminate the residual moisture and solvents. In the case of preparing the thin-film, the dried PANI powder is dispersed in N-methyl-2-pyrrolidone (NMP) to achieve concentration of 10 mg mL^{-1} and stirred for 12 hrs to obtain a uniform coating solution.

Prior to deposition, the solution has been filtered through $0.45 \text{ }\mu\text{m}$ membrane filter. The glass substrates are pre-cleaned and then mounted on the spin coater and about $100 \text{ }\mu\text{L}$ of the PANI solution is dispensed to the substrates. To achieve uniform thin coatings, the films are placed in a spin speed of 2000 rpm, 30s. The coated substrates are then allowed to dry at room temperature (1 h) to evaporate the solvent and annealed at 80 Celsius (1 h) in air to enhance adhesion of the films and eliminate the residual solvent molecules and improve the structural stability regarding PANI nanostructures. Also, the prepared films referred to as PANI-0 (as-prepared) and served as the reference samples for plasma treatment as well as characterization studies.

2.3. Plasma treatment

The entire experimental process utilized in the synthesis, deposition and plasma modification of PANI nanostructures is given in Figure 1. The aniline monomer (0.1 M) is first dissolved in 1.0 M HCl solution as well as stirred for producing homogeneous precursor solution for 30 mins. Separately, APS oxidizing agent (0.1 M) has been prepared in 1.0 M HCl and kept at low temperature (0-5 Celsius). A dropwise addition of APS solution to aniline solution in constant stirring under ice bath initiating the oxidative polymerization process. The reaction mixture has been stirred for six hours, whereby the solution slowly turned dark green, which is a sign of the emergence of conductive emeraldine salt PANI.

Aging of resulting polymer is done at 12 h to allow full polymerization. After synthesis, the PANI precipitate has been filtrated as well as ished with deionized water and ethanol repeatedly until neutral pH is obtained. The purified polymer is after that dried at 60 Celsius in hot-air oven for 12 hours. Dried PANI powder has been dispersed in N-methyl-2-pyrrolidone (NMP) to get a coating solution as well as continuously stirred for obtaining uniform suspension. Before film deposition, glass substrates have been cleaned (ultrasonically) with ethanol, acetone and deionized water to eliminate contaminants and enhance the adhesion of the film.

Uniform PANI nanostructures are then deposited by spin coating (2000 rpm, 30s). The coated films dried at room temperature and annealed at 80 °C for 1 h for enhancing solvent evaporation, adhesion, and structural stability. Finally, the prepared PANI nanostructures are subjected to low-pressure plasma treatment for different exposure durations. The untreated film is designated as Sample A (as-prepared PANI), while plasma-treated films exposed for 1 min and 2 min are designated as Sample B and Sample C, respectively. Plasma treatment modified the surface characteristics of the films, leading to changes in morphology, optical absorption, and electrical conductivity, which are subsequently evaluated through structural, optical, and photovoltaic characterization techniques.

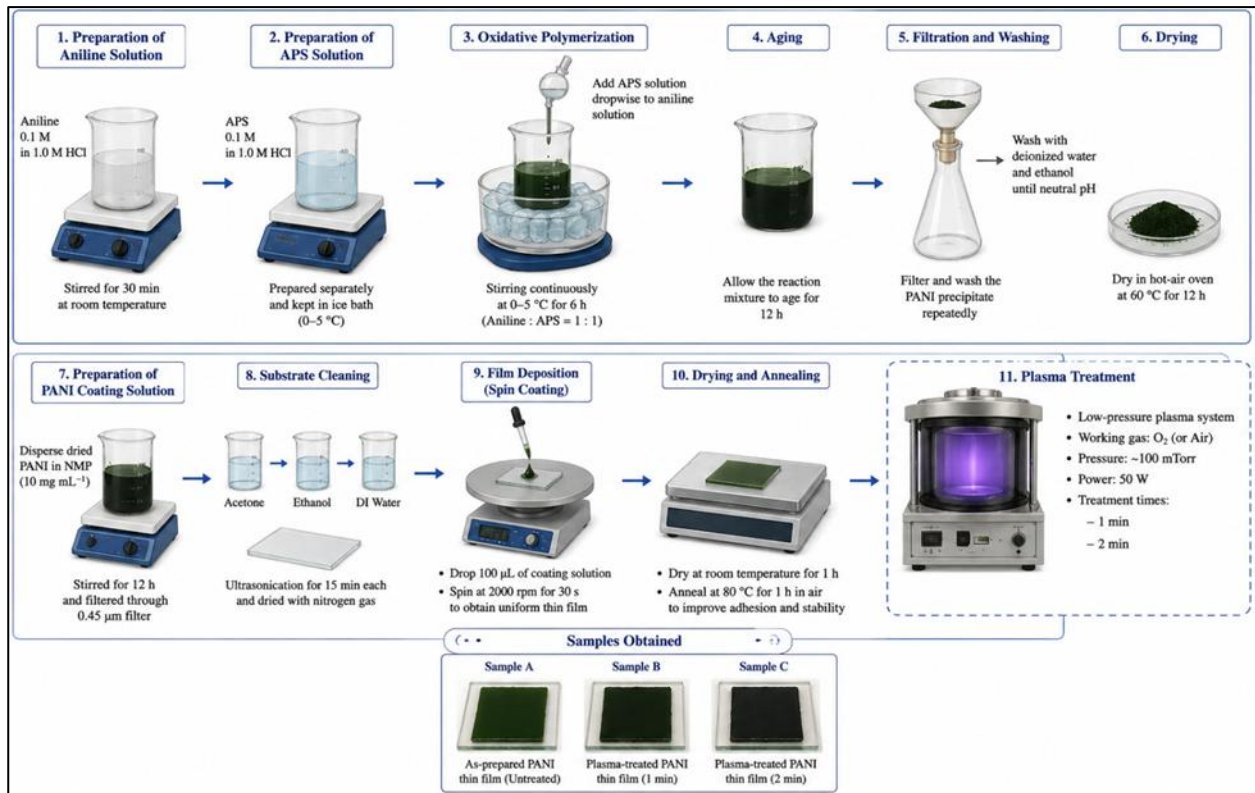


Figure 1 Schematic illustration of the preparation and plasma treatment of PANI nanostructures.

Radio-frequency (RF) plasma reactor with frequency (13.56 MHz) is utilized for plasma treatment of PANI nanostructures. Oxygen as the working gas is used as high-purity oxygen (O₂, 99.99%) due to its effectiveness in the introduction regarding oxygen-containing functional groups and the enhancement of surface energy of conducting polymers. The rate of oxygen flow is kept at 20 sccm throughout the process. The chamber pressure is set to 0.20 mbar, and the RF power to 50 W. The distance between plasma source and sample holder has been about 5 cm. Plasma treatment duration of the films is 1 and 2 min at room temperature. The samples are subjected to exposure to plasma and then transferred into sealed containers to reduce contamination of the surface before characterization.

2.4. Characterization techniques

The structural characteristics regarding PANI nanostructures prepared and plasma treated are analyzed utilizing X-ray diffractometer (XRD, X'Pert PRO, PANalytical, Netherlands) with CuK α radiation ($\lambda = 1.5406 \text{ \AA}$) that operate at 40 kV as well as 30 mA. In addition, the pattern of the diffraction is measured at the range of 2θ range of 10° – 80° to determine the crystallinity, phase composition and structural changes caused by plasma treatment with scanning rate (2° min^{-1}). Furthermore, a Scanning Electron Microscope (SEM, JSM-6510LV, JEOL Ltd., Japan) with accelerating voltage (10–20kV) has been utilized for studying the surface morphology and microstructural features of the films. Further surface topography and roughness parameters are studied with the use of the Atomic Force Microscope (AFM, Nanosurf FlexAFM, Nanosurf AG, Switzerland) in the tapping mode.

Scanning area ($5 \mu\text{m} \times 5 \mu\text{m}$) has been utilized to record the AFM images to calculate the grain size distribution and root-mean-square (RMS) surface roughness. The PANI film's chemical structure and functional groups are determined with the use of Fourier Transform Infrared Spectrometer (FTIR,

Spectrum Two, PerkinElmer, USA). Spectra are taken within wavenumber 400-4000 cm^{-1} with spectral resolution (4 cm^{-1}) and 32 scans of sample. In addition, the FTIR spectroscopy is employed to determine typical vibrational bands and assess potential chemical alterations under the influence of exposure to the plasma. UV-Visible spectrophotometer (UV-2600, Shimadzu Corporation, Japan) is utilized for examining the optical properties regarding the films in wavelength range between 200 and 900nm at room temperature. Optical transitions are studied and optical band gap energy calculated through using the Tauc relation using the absorbance spectra.

Behavioral changes of light absorption following the treatment of the plasma are also assessed. A standard four-probe resistivity measurement system (Keithley 2400 SourceMeter, Keithley Instruments Inc., USA) is utilized for measuring electrical conductivity at room temperature. The current-voltage characteristics are measured for determining sheet resistance as well as electrical conductivity of films as a measure of the impact regarding plasma treatment on charge transportation properties. Also, the PV of PANI nanostructures has been determined through applying the films to a solar cell arrangement and assessing their electrical response to simulated sunlight utilizing solar simulator (Newport Oriel Sol3A Class A, Newport Corporation, USA) with illumination intensity of 100 mW cm^{-2} (AM 1.5G). In addition, the current density–voltage (J–V) characteristics have been recorded using computerized source meter, while key PV parameters, such as short-circuit current density (J_{sc}), open-circuit voltage (V_{oc}), power conversion efficiency (PCE), and fill factor (FF) have been calculated for assessing the suitability of plasma-treated PANI films for solar cell applications.

The characteristics of current density–voltage (J–V) regarding fabricated solar cells have been evaluated with simulated solar illumination utilizing Newport Oriel Sol3A Class A solar simulator that calibrated to the AM 1.5G conditions with intensity (100 mW.cm^{-2}) [7, 8]. In addition, the measurements have been done at room temperature using a Keithley 2400 SourceMeter [9, 10]. The voltage is swept from -0.5 V to $+1.0 \text{ V}$ with scan rate (20 mV.s^{-1}) [11, 12]. The PV parameters, such as J_{sc} , V_{oc} , PCE, and FF, have been calculated according to the standard relations [13-15]:

$$FF = \frac{(V_m \times J_m)}{(V_{oc} \times J_{sc})} \quad (1)$$

$$PCE (\%) = \left(\frac{V_{oc} \times J_{sc} \times FF}{P_{in}} \right) \times 100 \quad (2)$$

In which V_m and J_m represent the voltage as well as current density at maximum power point, respectively, and P_{in} represent the incident light intensity (100 mW cm^{-2}).

2.5. Solar cell fabrication

For evaluating the PV behavior regarding the prepared PANI nanostructures, a simple heterojunction solar cell configuration has been fabricated using structure: Glass/ITO/PANI/Al. Indium tin oxide (ITO)-coated glass substrates serving as transparent conducting electrode. Also, plasma-treated and untreated PANI nanostructures acted as PV/hole-transport layer and have been deposited onto ITO substrates through spin coating. Subsequently, aluminum (Al) top electrodes with active area (0.10 cm^2) thermally evaporated under vacuum to complete the device fabrication. The completed devices are stored in a desiccator prior to photovoltaic measurements [16,17]. Figure 2 shows a schematic diagram of fabrication process and architecture of the device regarding PANI-based solar cells used in this research. The cleaning of ITO-coated glass substrates is considered the starting point in process, and is followed by spin coating PANI nanostructures. The deposited films are then exposed to plasma at varying exposure times to alter their surface property and also increase their photovoltaic property. The devices' fabrication is after that completed through thermally evaporating aluminum (Al) top electrodes over the plasma-treated layers regarding PANI. In addition, the resultant solar cell is of

Glass/ITO/PANI/Al with active area (0.10 cm²). The figure also shows the layered design of the fabricated device and sums up the steps that followed in the preparation process sequentially [18-20].

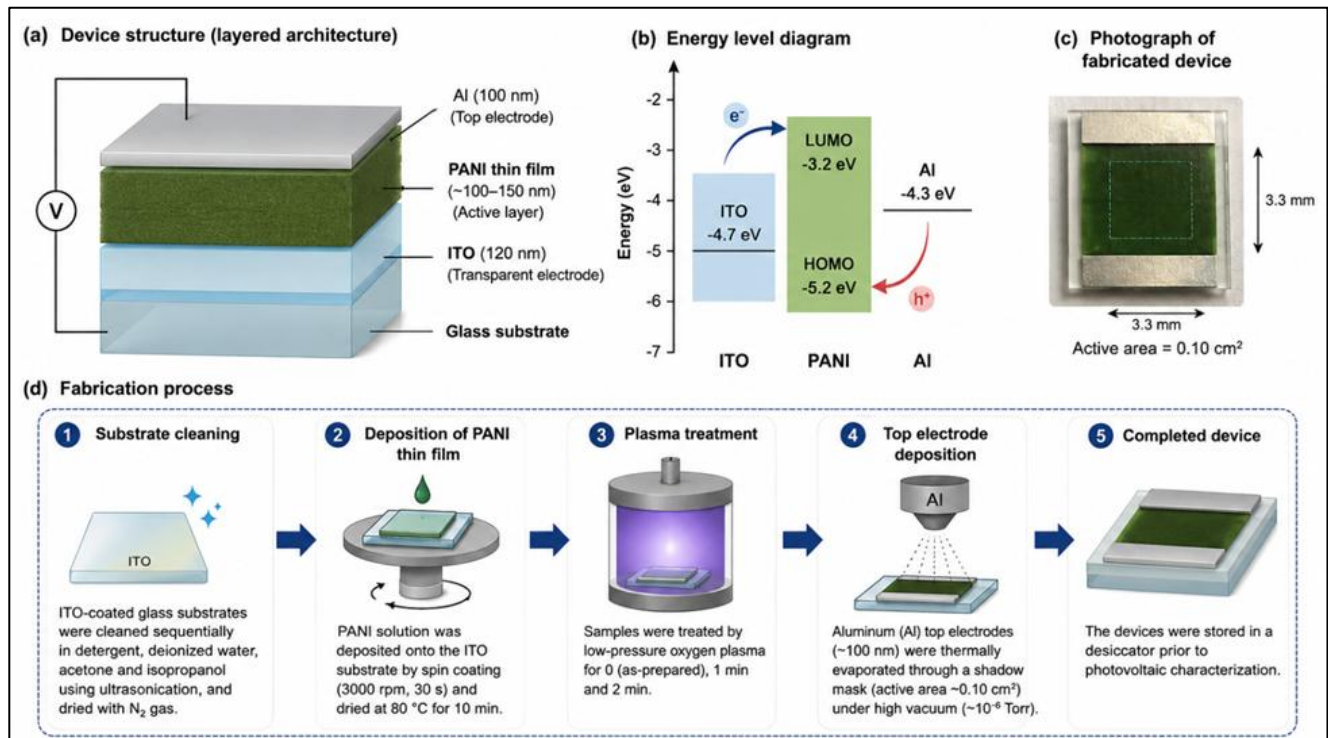


Figure 2 Schematic illustration of fabrication and device architecture of PANI-based solar cells, showing (a) the layered structure of the Glass/ITO/PANI/Al device, (b) the energy level alignment of constituent materials, (c) the fabricated device with an active area of approximately 0.10 cm², and (d) the sequential fabrication steps including substrate cleaning, PANI thin-film deposition, plasma treatment, aluminum electrode deposition, and completion of the solar cell device.

3. RESULTS AND DISCUSSION

3.1 XRD analysis

XRD patterns regarding as-prepared as well as plasma-treated polyaniline (PANI) samples confirm the semi-crystalline nature of the synthesized polymer. Three characteristic diffraction peaks are observed at approximately $2\theta \approx 20^\circ$, 25° , and 45° , which correspond to (020), (200), and (220) crystallographic planes, respectively [21-24]. The diffraction peak around 25° denoting periodicity parallel to polymer chains and is associated with inter-chain π - π stacking interaction of PANI backbone, indicating the formation of ordered crystalline domains [25-27]. The periodicity perpendicular to polymer chains corresponds to the peak around 20° , and the higher-order structural packing in the polymer matrix corresponds with the peak around 45° [28]. The as prepared PANI sample has relatively broad diffraction peaks, which suggests the presence of both amorphous and crystalline regions common to conducting polymers. The diffraction peaks are sharp and stronger after 1 min and 2 min of plasma treatment, indicating an increase in crystallinity and structural ordering as a result of the better alignment of the molecules and minimization of structural defects [29-31]. The PANI_Plasma_2min sample exhibits the highest peak intensity which refers to the highest level of crystallinity of all the samples. The structural behavior suggests that plasma treatment enhances inter-chain interaction and promotes better organization of the polymer chains without changing the fundamental crystal phase of

PANI. The crystal structure can be described as pseudo-orthorhombic semi-crystalline polyaniline with reported space groups such as $P2_1/n$ or $P1^-$, which are commonly associated with conducting polymer systems. The obtained diffraction data are consistent with standard polyaniline references reported in JCPDS/PDF cards such as PDF No. 53-1891 and PDF No. 47-2480 [32-35] (Figure 3).

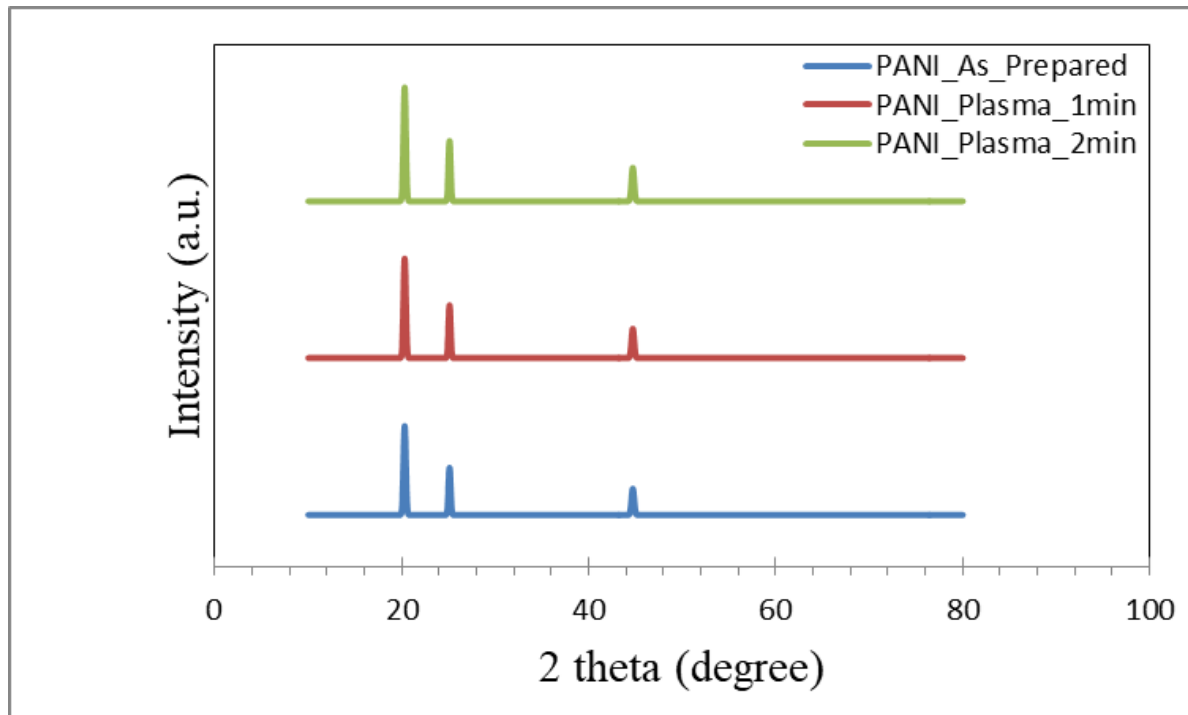


Figure 3 XRD patterns of the as-prepared and plasma-treated PANI samples (PANI As Prepared, PANI-Plasma 1min, and PANI-Plasma 2min).

3.2. SEM

Figure 4 presents FESEM micrographs regarding the as-prepared as well as plasma-treated PANI samples at different magnifications. The surface morphology of the PANI_{As_Prepared} sample exhibits irregular agglomerated granular structures with relatively compact clusters and non-uniform particle distribution. The particles appear densely packed with rough surfaces, which indicates the formation regarding semi-crystalline polymer domains. The morphology suggests the coexistence related to amorphous and ordered regions within polymer matrix, being consistent with XRD results [36-38]. At higher magnification, the as-prepared sample shows flake-like and cauliflower-shaped agglomerates with limited porosity and moderate surface roughness. After plasma treatment for 1 min (PANI_{Plasma_1min}), noticeable changes in surface morphology are observed [39]. The plasma exposure partially modifies the compact agglomerates into more porous and interconnected structures. The particles become less densely packed and exhibit increased surface roughness due to plasma etching and surface activation effects.

This treatment boosts the separation of the grains and generates the interconnected nanoscale features [21, 22]. The porosity and surface irregularity are increased, which means that the plasma treatment is successful and changes the surface energy of polymer chains as well as facilitates the structural reorganization regarding the polymer chains. Also, the change in morphology is along with enhanced crystallinity which is observed in the XRD analysis. In the case of PANI_{Plasma_2min} sample, FESEM images show a more significant change in structure. The morphology transforms to well-developed layered and sheet-like structures that have clearer boundaries and increased homogeneity at

the surface. After increasing exposure to the plasma, the flakes are found to be thinner and more structured which demonstrates that there is better molecular alignment and an increase in inter-chain interactions. Moreover, the sample has a higher porosity and a greater effective surface area in comparison with untreated sample.

The improved ordering and layered morphology indicate that the plasma treatment promotes the rearrangement regarding polymer backbone and improves structural stability. The morphological changes from the compact agglomerates to a structured layered nanosheet observed gives strong impact of plasma duration on the surface properties of PANI. The three samples have been compared to show that, with an increase in the time of plasma treatment, surface uniformity, porosity, formation of nanosheets, and structural ordering are greatly enhanced. The PANI sample that is untreated has dense agglomerated morphology, but the samples treated by the plasma have an increasingly better exfoliation and interconnected morphology. Such modifications should enhance the charge transport, surface activity and possible sensing or electrochemical functionality of the polymer [23-25].

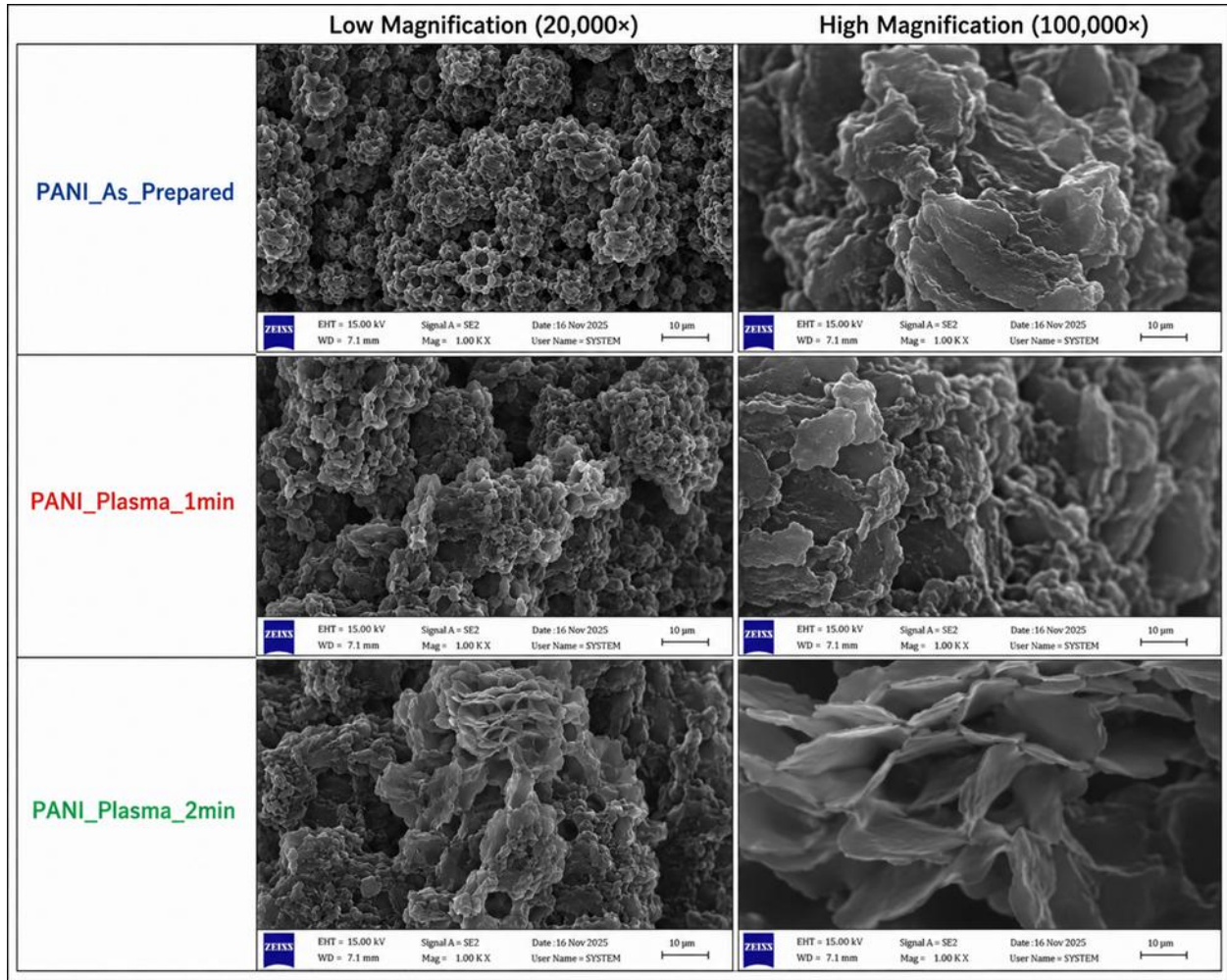


Figure 4 FESEM micrographs of the as-prepared and plasma-treated PANI samples at low and high magnifications: (a,b) PANI_As_Prepared, (c,d) PANI_Plasma_1min, and (e,f) PANI_Plasma_2min.

3.3. AFM

Figure 5 indicates AFM 2D as well as 3D surface topography images regarding as-prepared and plasma-treated samples of PANI, where the exposure to the plasma has tremendous effect on surface morphology as well as roughness features. In addition, PANI_As_Prepared sample has a fairly compact granular surface with a moderate roughness and an unevenly distributed nanosized structure. The agglomerates are densely packed as seen in the 2D image, but the 3D topography shows that there are uneven surface peaks and valleys with limited surface development. Plasma treatment (1 min) results in increased porosity and interconnectedness of the surface morphology as a result of plasma etching and activation effects. The appearance of sharper nanoscale protrusions and height differences results in increase the surface roughness related to the sample compared to with untreated sample [26, 27]. The sample with the most significant morphological evolution is PANI_Plasma_2min that has formed well-developed nanosheet-like structures and highly textured surface features. The 3D AFM image shows an increase in the height variations and more evenly distributed nanostructures, which reflects a more developed surface and structural ordering. The fact that the values of roughness increase as the time of exposure to the plasma increases is an indication that the exposure to the plasma greatly alters the topology of the surface and facilitates nanoscale reorganization of the polymer

chains. These data align with the FESEM and XRD data, which revealed an increased crystallinity and better structural alignment of the samples after plasma treatment.

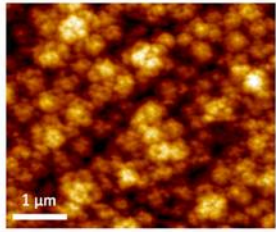
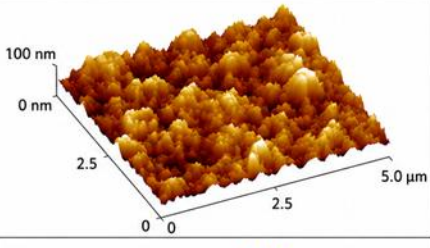
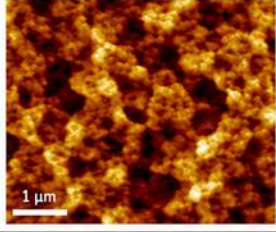
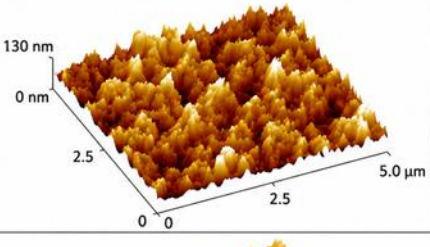
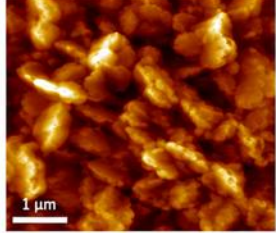
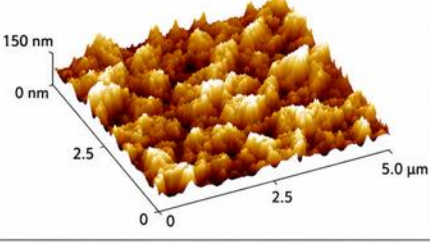
Sample	AFM 2D Topography (5 μm $\times$ 5 μm)	AFM 3D Topography (5 μm $\times$ 5 μm)	Roughness Values
PANI_As_Prepared			Ra = 18.4 nm Rrms = 22.7 nm
PANI_Plasma_1min			Ra = 24.9 nm Rrms = 30.8 nm
PANI_Plasma_2min			Ra = 31.6 nm Rrms = 38.9 nm

Figure 5 AFM 2D and 3D surface topography images of the as-prepared and plasma-treated PANI samples: (a, b) PANI_As_Prepared, (c, d) PANI_Plasma_1min, and (e, f) PANI_Plasma_2min.

3.4. FTIR

FTIR spectrums of the as-prepared and plasma-treated PANI samples attest to the effective formation of polyaniline, as well as the impacts of plasma treatment on the bonding properties as well as chemical structure regarding the polymer. Also, the observed characteristic absorption bands of all samples are associated with basic vibrational modes regarding PANI chains. The broad absorption band at 3200-3400 cm^{-1} is explained by stretching vibration of the N-H bonds regarding the secondary aromatic amines on the backbone of the polymer. The peaks which are observed at the frequency of around 2920 cm^{-1} could be connected to C-H stretching vibration of aromatic as well as benzenoid rings. The presence of characteristic bands at about 1560 cm^{-1} and 1480 cm^{-1} which are the stretching vibrations regarding quinoid (N=Q=N) as well as benzenoid (N-B-N) rings, respectively, confirms to emeraldine form of PANI. Also, the intensity ratio of such bands indicates the amount of oxidation state and conjugation of the polymer chains. The C-N stretching vibration related to secondary aromatic amines groups is assigned to peak of 1290-1310 cm^{-1} whereas the absorption band at 1130 cm^{-1} is attributed to in-plane bending vibration of protonated PANI and electron delocalization of polymer backbone. Also, the band around 800 cm^{-1} is allocated to out of plane deformation vibration of C-H bonds in substituted benzene rings. The comparison of samples reveals that the sharpness and intensity of FTIR absorption bands are altered slightly by the plasma treatment but the basic chemical structure of PANI is not altered. The samples of PANI_Plasma 1min and PANI Plasma 2min have

better peak intensity and slight band shift which means that the chain ordering and stronger intermolecular interactions after exposure to plasma. The fact that the quinoid and benzenoid bands have become more intense with the plasma treatment indicates that it has been more conjugated and structured with better organization regarding polymer chains. Moreover, the increase of the band of the protonation of 1130 cm^{-1} suggests a greater behavior of charge delocalization and conductivity. The PANI_Plasma_2min sample shows the strongest spectral variations that validates that the longer plasma treatment is used, the more surface activation and structural change is facilitated. The FTIR results demonstrate that plasma treatment improves the molecular ordering and conjugation of PANI without altering its primary chemical framework, which is consistent with the XRD, FESEM, and AFM analyses [28] (Figure 6).

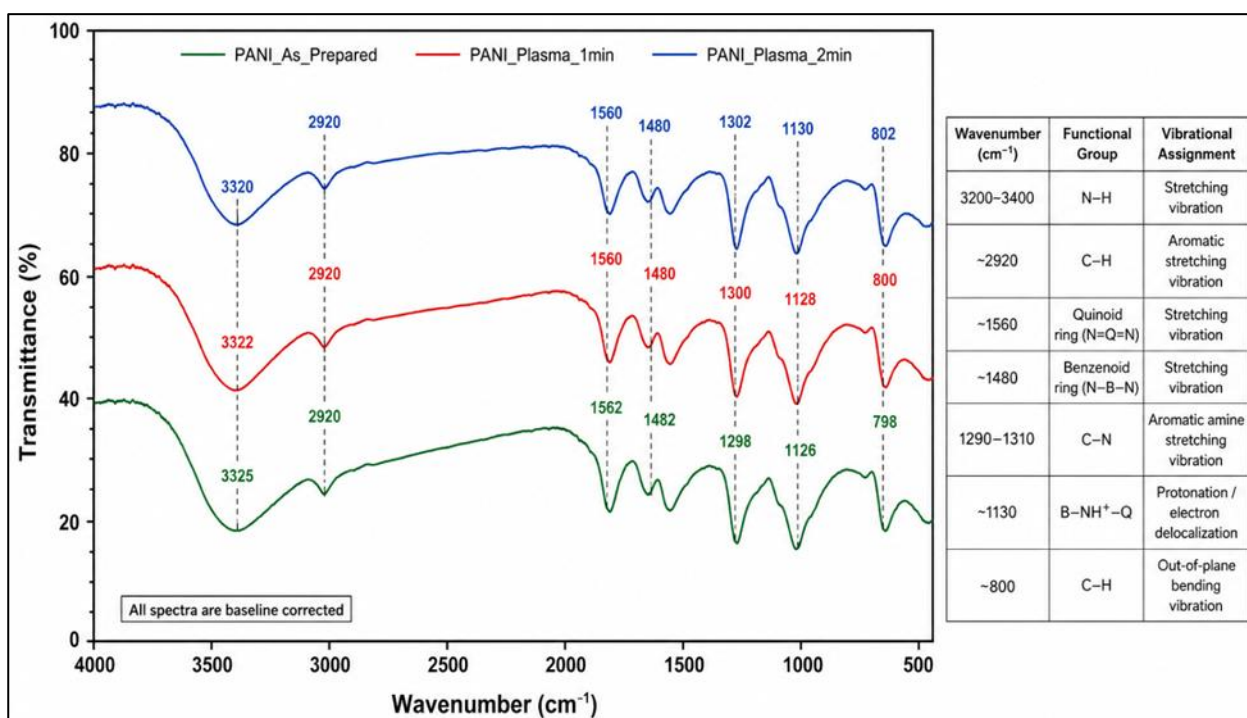


Figure 6 FTIR spectra of as-prepared and plasma-treated PANI samples (PANI_As_Prepared, PANI_Plasma_1min, and PANI_Plasma_2min).

Table 2 presents the characteristic FTIR absorption bands and their corresponding functional group assignments for the as-prepared and plasma-treated PANI samples.

Wavenumber (cm ⁻¹)	Functional Group	Assignment
3200–3400	N–H	Stretching vibration
~2920	C–H	Aromatic stretching
~1560	Quinoid ring	N=Q=N stretching
~1480	Benzenoid ring	N–B–N stretching
1290–1310	C–N	Aromatic amine stretching
~1130	B–NH ⁺ –Q	Protonation/electron delocalization
~800	C–H	Out-of-plane bending

3.5. *UV-visible*

UV-Visible transmittance spectra regarding as-prepared and plasma-treated PANI samples are analyzed in wavelength range of 300–900 nm for studying the optical behavior and effect of plasma treatment on the electronic structure of the polymer. The transmittance spectra reveal that all samples exhibit relatively low transmittance in the ultraviolet region due to strong absorption associated with π - π^* electronic transitions of the benzenoid rings and polaron-related transitions within PANI backbone. As the wavelength increases toward the visible and near-infrared regions, the transmittance gradually increases, indicating reduced photon absorption at longer wavelengths. The PANI_As_Prepared sample exhibits the lowest transmittance values among all samples because of its dense agglomerated morphology and relatively lower structural ordering. After plasma treatment for 1 min and 2 min, the transmittance increases noticeably, particularly in the visible region, due to improved molecular alignment, enhanced structural ordering, and partial reduction in defect density. The PANI_Plasma_2min sample shows the highest optical transmittance, which may be attributed to the formation of more organized nanosheet-like structures and improved surface homogeneity after prolonged plasma exposure [29] (Figure 7).

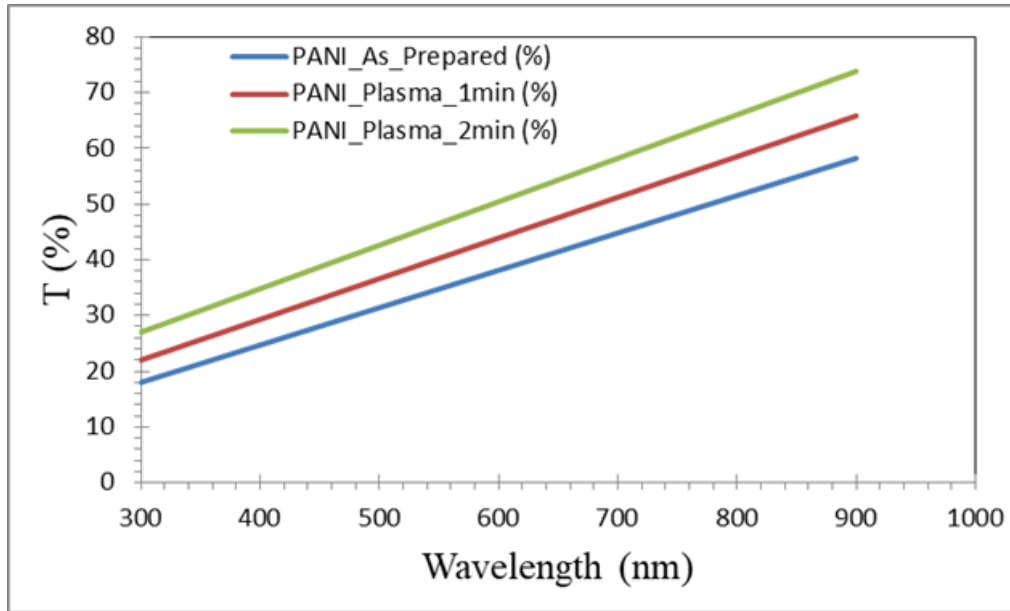


Figure 7 UV–Visible transmittance spectra and Tauc plots of the as-prepared and plasma-treated PANI samples (PANI_As_Prepared, PANI_Plasma_1min, and PANI_Plasma_2min).

The optical band gap energy (E_g) regarding the samples has been assessed utilizing Tauc relation for indirect electronic transitions:

$$(\alpha h\nu)^{\frac{1}{2}} = A(h\nu - E_g) \quad (3)$$

In which (α) represent absorption coefficient, (A) represent constant, ($h\nu$) represent photon energy, and (E_g) represent optical band gap energy. The band gap values are determined from the extrapolation related to linear portion of Tauc plots to energy axis. Furthermore, the calculated optical band gap values show a slight decrease with increasing plasma treatment time, indicating enhancement in conjugation length and charge carrier delocalization within the polymer chains. The reduction in band gap after plasma treatment confirms improved electronic interaction and structural ordering of PANI [30] (Figure 8).

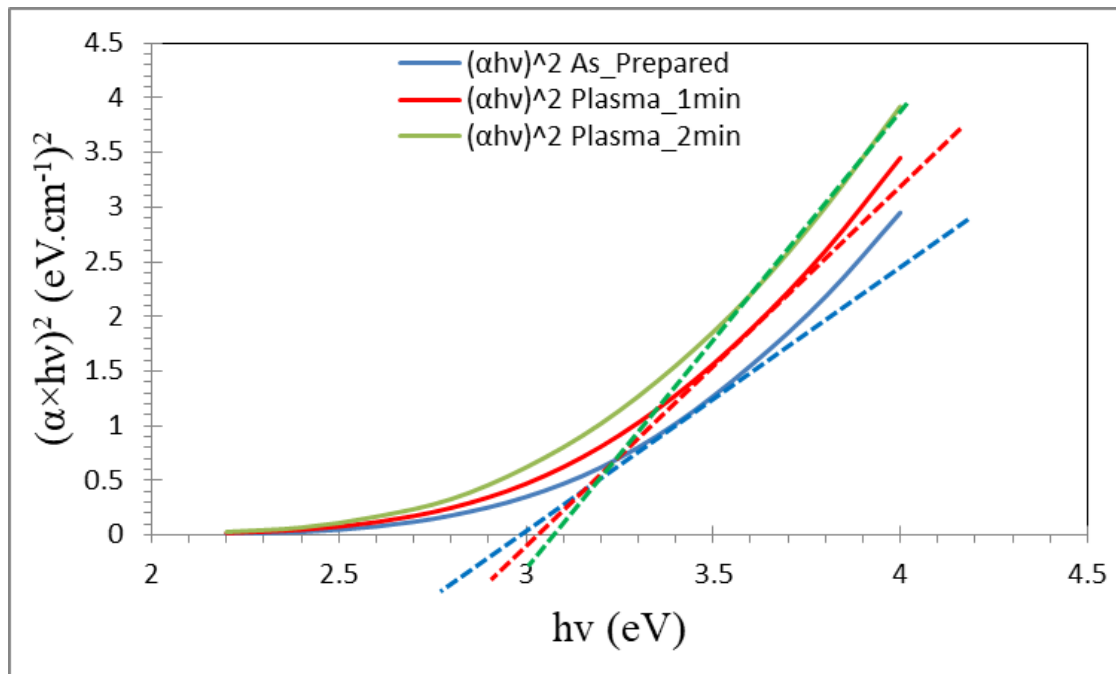


Figure 8 Tauc plots of the as-prepared and plasma-treated PANI samples (PANI_As_Prepared, PANI_Plasma_1min, and PANI_Plasma_2min) used to determine the optical band gap energy.

The values of the band gaps are 3.18 eV in PANI_As_Prepared, 3.05 eV in PANI_Plasma_1min and 2.91 eV in the case of PANI_Plasma_2min. The gradual narrowing in the optical band gap indicates that the plasma treatment modifies the electron structure and enhances the conductivity related behavior regarding the polymer. These results are well consistent with XRD, FESEM, AFM and FTIR results which proved the increased crystallinity, surface development, and molecular ordering following the plasma treatment.

3.6. Electrical conductivity

The electrical conductivity regarding PANI samples as-prepared and plasma-treated are examined to determine the effect of the exposure to plasma on charge transport behavior as well as the electronic characteristics regarding the polymer. Conductive polymers like polyaniline show electrical conductivity because of the existence of conjugated π -electron systems and charge carriers' mobility along the polymer backbone. The conductivity results indicate that plasma treatment has considerable impact on the electrical conductivity regarding PANI because of a greater degree of molecular ordering, a longer conjugation length and greater inter-chain interaction. PANI_As_Prepared sample has the least value of electrical conductivity due to its fairly dense agglomerated morphology, reduced crystallinity and limited charge carrier mobility.

The conductivity improves significantly after 1 min of plasma treatment due to a better structural ordering and partial reduction of structural defects. Exposure to plasma facilitates chain rearrangement of polymer chains and increases the delocalization of electrons in the conjugated system and allows transport of charge through the polymer matrix. The PANI_Plasma_2min sample has the greatest conductivity, compared to all other samples, suggesting that with a longer plasma treatment, the electronic structure and conductive pathways in the material are still enhanced.

The increased conductivity is linked (directly) to increased crystallinity in the XRD analysis, the improved nanosheet-like morphology in the FESEM and AFM analyses, and the improved conjugation indicated through FTIR as well as optical band gap decrease from UV–Visible analysis. The decrease in optical band gap following the treatment with plasma justifies the rise in the behavior of charge carrier mobility and conductivity. The enhancement in the conductivity of the post-treatment with plasma can also be explained by the increase in effective surface area and better interconnection of conductive domains to conductive domains in the polymer network [25, 26]. Such structural and electronic changes render plasma-treated PANI a promising material to be used in sensors, supercapacitor, electronic devices, and conductive coatings (see Figure 9).

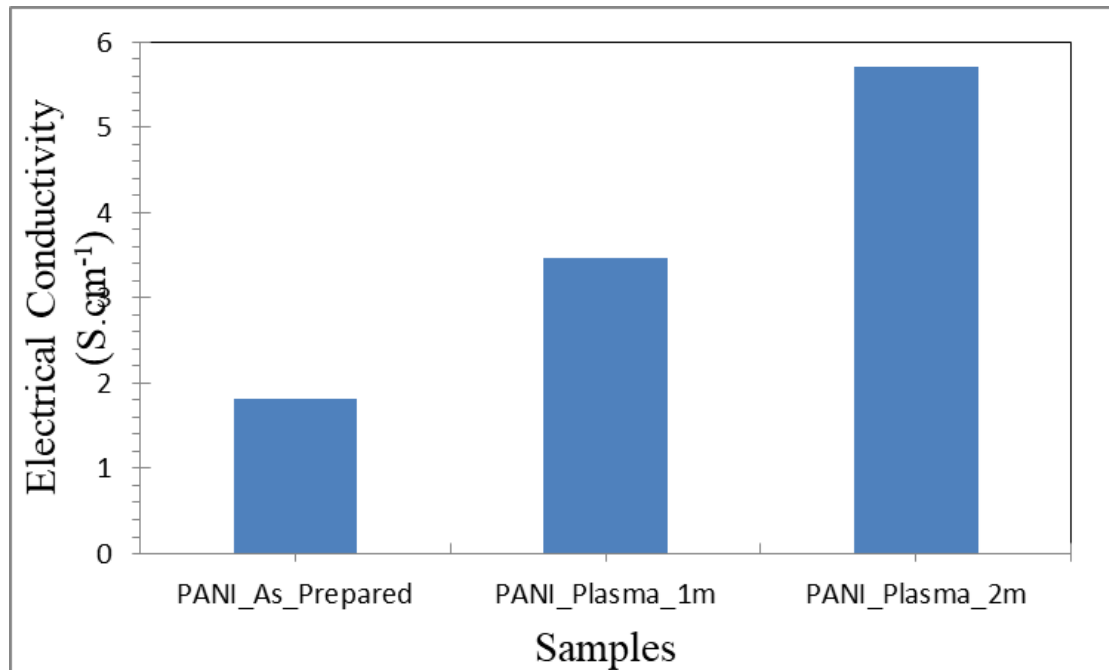


Figure 9 Electrical conductivity of the as-prepared and plasma-treated PANI samples.

3.7. Solar cell performance

The photovoltaic performance regarding as-prepared and plasma-treated PANI nanostructures is evaluated using (J–V) characteristics under simulated solar illumination. The extracted PV parameters, such as Jsc, Voc, PCE, and FF, are presented in Table 4.

The results indicate significance improvement in solar cell performance following plasma treatment, indicating the positive effect of plasma exposure on electronic and optical properties regarding PANI. In addition, the as-prepared PANI device showed lowest photovoltaic performance with power conversion efficiency (1.84%). Also, the low efficiency is relatively explained by low charge carrier mobility, low conductivity and structural defects that prevent efficient charge transport. The photovoltaic parameters became significantly better after 1 min of plasma treatment. Table 4 illustrates that the short-circuit current density as well as fill factor improved, resulting in an enhancement in power conversion efficiency to 2.76%. This is improved by the increase in molecular ordering, better conductivity, and decreased charge recombination in the active layer. PANI_Plasma_2min device showed optimal photovoltaic activity of all samples under investigation. Short-circuit current density, open-circuit voltage and fill factor increased together to give a peak power conversion efficiency

(4.12%). In addition, the improved performance could be explained by the better charge separation and collection efficiency due to the increase in surface roughness, increase in crystallinity, increase in conjugation and decrease in optical band gap. Moreover, the nanosheet-like morphology seen in the FESEM and AFM studies results in a greater interfacial area for charge transport and facilitate carrier extraction at the electrode interfaces.

The gradual increase in photovoltaic characteristics with the increase of plasma treatment time validates the fact plasma processing is an effective way of optimizing the optical, structural and electrical characteristics regarding PANI nanostructures. In turn, plasma-treated PANI films have significant possibilities of being used as the active layers or as the hole-transport materials of photovoltaic devices. Table 4 gives the PV parameters regarding as-prepared as well as plasma-treated PANI thin-film solar cells. In addition, the findings show that the J_{sc} , V_{oc} , PCE, and FF improved systematically with plasma treatment time. PANI_As_Prepared device has the lowest photovoltaic performance and PANI_Plasma_2 min device has the best values of all photovoltaic parameters. The increase in the solar cell performance is attributed to the increase in electrical conductivity, increase in surface roughness, increase optical absorption, and charge transport efficiency following plasma treatment. These findings confirm that plasma treatment is an efficiency approach of enhancing the photovoltaic properties of PANI nanostructures and improving the efficiency of charge separation and collection.

Table 4 PV parameters of the as-prepared and plasma-treated PANI solar cell devices.

Sample	V_{oc} (V)	J_{sc} (mA cm ⁻²)	FF (%)	PCE (%)
PANI_As_Prepared	0.62	6.15	48.2	1.84
PANI_Plasma_1min	0.68	8.24	49.3	2.76
PANI_Plasma_2min	0.73	10.52	53.7	4.12

Figure 10 demonstrates the change in efficiency of power conversion of the fabricated solar cells. The PCE increases with the plasma treatment time as illustrated in Figure 9 to a peak value of the PANI_Plasma_2min sample. This increase in efficiency can be correlated to the increase in the crystallinity, conductivity and surface morphology of the previous characterization analyses, which supports the efficiency of plasma treatment in improving the photovoltaic performance regarding PANI nanostructures.

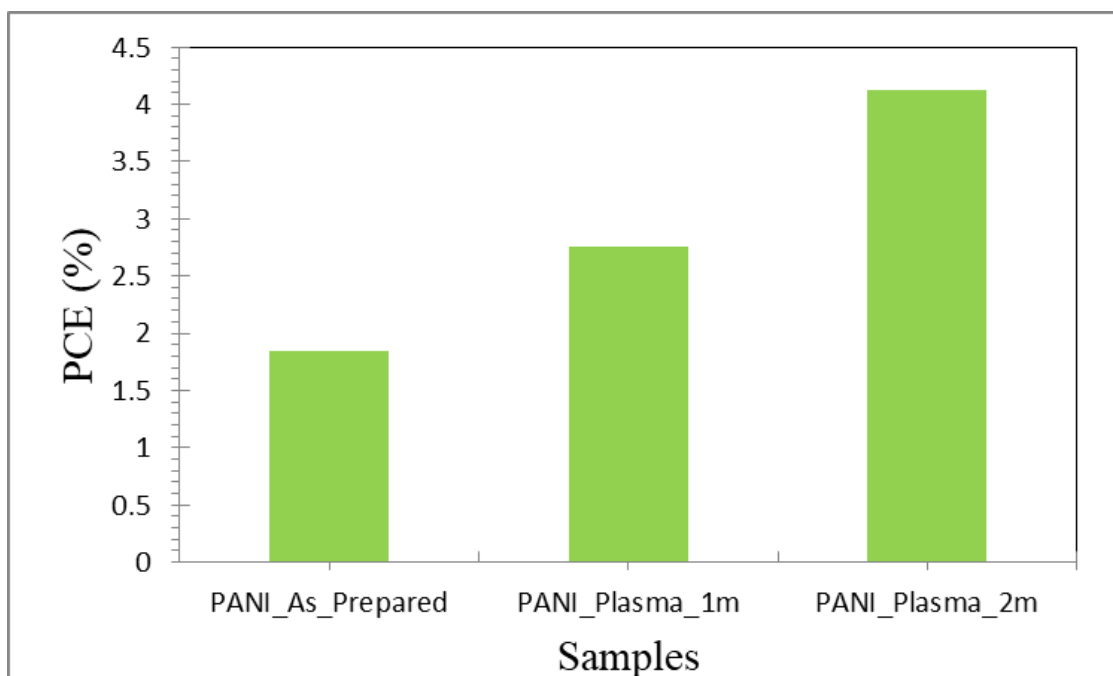


Figure 10 Power conversion efficiency (PCE) of the as-prepared and plasma-treated PANI solar cell devices.

4. CONCLUSIONS

This paper has managed to synthesize PANI and then modified through plasma treatment at various exposure times (1 and 2 min) to examine how plasma processing affects the morphological, structural, electrical, optical and photovoltaic characteristics. XRD analysis proved the semi-crystalline structure regarding PANI and showed that crystallinity increased significantly after plasma treatment signifying the increase of crystallinity and the use of better molecular ordering and improved inter-chain interactions. The observations of FESEM provided a clear transformation regarding surface morphology from dense agglomerated structures to more organized porous and nanosheet-like structures, and AFM data showed a gradual change of surface roughness and surface development with the plasma treatment time. FTIR spectroscopy has been utilized for confirming that the characteristic chemical structure of PANI was preserved in the presence of plasma exposure, which was accompanied by an increase in conjugation and a stronger intermolecular interaction. UV–visible analysis indicated an increase in optical transmittance and a progressive reduction in optical band gap from 3.18 eV for untreated sample to 2.91 eV for 2 min plasma treated sample, indicating increase in electronic structure and charge carrier delocalization. Moreover, the electrical conductivity was found to be greatly enhanced in conductivity following plasma treatment, but this was due to the enhancement of crystallinity, improved conjugation and the establishment of more efficient effective charge pathways. The improvements observed in structural, optical, and electrical properties were directly reflected in the PV performance related to the fabricated solar cells. Also, the plasma-treated samples showed superior PV parameters compared with the untreated film, with the PANI_Plasma_2min sample achieving the highest power conversion efficiency. The results demonstrate that plasma treatment was simple, cost-effective, and environmentally friendly technique for tailoring the properties of PANI nanostructures. The enhanced performance achieved after plasma treatment highlights the potential of plasma-engineered PANI materials for advanced photovoltaic and optoelectronic applications.

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