



Effect of Dielectric Barrier Discharge (DBD) Plasma on Structural and Electrical Properties of Polypyrrole (PPy) Thin Films

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Abstract

Cold plasma treatment is an effective approach for surface modification of conducting polymers, enabling the tuning of their structural and electronic properties without altering the bulk composition. In this work, polypyrrole (PPy) thin films were fabricated on indium tin oxide (ITO) substrates via electrochemical deposition. The films were subsequently exposed to dielectric barrier discharge (DBD) cold plasma for different exposure times (0, 5, 10, 15, and 20 min). The structural modifications induced by plasma treatment were investigated using X-ray diffraction (XRD), while the electrical and charge transport properties were analyzed using Hall effect measurements. The XRD results revealed significant changes in peak intensity and crystallinity, indicating surface activation and partial reorganization of polymer chains upon plasma exposure. Hall effect measurements showed systematic variations in carrier concentration, mobility, and electrical conductivity as a function of plasma treatment time, confirming the strong influence of surface modification on charge transport behavior in PPy films. The results demonstrate that DBD cold plasma treatment provides a controllable and effective route for enhancing the structural ordering and electrical performance of electrochemically deposited PPy thin films, making them promising for applications in sensors, optoelectronic devices, and flexible electronics.

Keywords: Cold plasma, Polypyrrole, X-ray diffraction, Hall effect.

1. Introduction

Polymers, by their structural properties, possess the ability to act as electrical insulators. On the other hand, the possibility of modifying their physical and chemical properties to reverse their conductive properties and produce conductive polymers has been studied for many years. In most cases, studies have focused on the manufacture of conductive polymers containing elements that improve their electrical properties. Polypyrrole (PPy) is a polymer used in many technological applications as a semiconductor due to its excellent electrical conductivity and relatively good thermal stability. It has distinctive properties and is highly susceptible to the presence of other elements or compounds in its structure, whether by chemical impurities, ion implantation, copolymerization, or as a metal complex. Polypyrrole has been actively used in many applications in microprocessors and bio-based applications and has been synthesized by various techniques such as electrochemical oxidation and chemical oxidation of pyrrole monomer and also, Polypyrrole (PPy) is utilized in the fabrication of solid-state electronic devices, including metal-insulator-semiconductor FET structures and metal-semiconductor configurations, due to its diverse range of energy band gaps¹⁻¹⁰. It consists of positively and negatively charged ions, electrons, and neutral entities (atoms and molecules), as well as radicals and ultraviolet rays. Plasma is categorized into two types: heat plasma and cold atmospheric plasma (CAP). Thermal plasma has heavy particles and electrons at an equivalent temperature, whereas Cold Atmospheric Plasma (CAP) features heavy particles

at a temperature lower than that of electrons, often below 40 °C, and remains at ambient temperature. Cold plasma has a reputation as one of the most effective tools in a wide range of applications. Non-thermal plasma applications have been subject to extensive studies.

Cold plasma has gained increasing interest because it offers scalable and environmentally friendly technical solutions even at atmospheric pressure and at a relatively low cost. One of the advantages of cold plasma is its ability to process thermally unstable samples (soft materials, organic materials, and polymers). And also, nonthermal plasmas have garnered significant interest due to their capacity to inactivate harmful microbes and pollutants, positioning them as a viable instrument for the eradication of chemical and biological contamination¹¹⁻¹⁹.

There are several methods for generating cold plasma from various gases, including dielectric barrier discharge (DBD), plasma jetting, slip arc discharge, and others. DBD is regarded as a standard method for producing a substantial volume of nonequilibrium air pressure diffuse plasma²⁰⁻²³.

The purpose of this research is to examine how the cold plasma treatment influences electrical and structural characteristics of polypyrrole films as a result of electrochemical deposition. To do that, the changes in the crystal structure are analyzed with X-ray diffraction (XRD), and the obtained results are supported by the analysis of the electronic variations and dynamics of inertness with the Hall effect test. Thus, the connection of the enhancement of electrical properties of polypyrrole and plasma surface modification, as has been shown in this research, makes it very useful in the electronic and sensor industry.

2. Materials and Methods

2.1. Materials

The chemicals used for the electrochemical deposition of polypyrrole (PPy) thin films were pyrrole monomer (C_4H_5N) as the primary precursor and potassium nitrate (KNO_3) as the supporting electrolyte. Indium tin oxide (ITO)-coated glass substrates were used as conductive electrodes. Deionized and distilled water were used throughout the preparation and cleaning processes. Cold plasma treatment was performed using a dielectric barrier discharge (DBD) system. The structural and electrical properties of the prepared films were investigated using X-ray diffraction (XRD) and Hall effect measurements.

2.2. Experimental Methods

The reaction solution was electrochemically precipitated. The preparation of the polypyrrole (PPy) film consisted of several steps using a 0.1 M pyrrole solution and 0.2 M potassium nitrate (KNO_3) as a supporting electrolyte. The molecular masses of the individual compounds were calculated based on their molar masses (potassium nitrate = 101.1 g/mol, pyrrole = 67.09 g/mol). The reagents were accurately weighed using a sensitive analytical balance.

The chemicals were dissolved in 100 mL of deionized water to prepare the stock solutions. The 0.1 M pyrrole solution was subsequently diluted to 0.07 M. The solution volume was adjusted using distilled/deionized water to ensure homogeneity. A mixed electrolyte solution was prepared by combining pyrrole and potassium nitrate solutions (6 mL each).

ITO-coated glass substrates were used as working electrodes. The substrates were cut into small pieces and cleaned by immersion in ethanol for 10 minutes, followed by rinsing with distilled water and drying with blotting paper. The conductive side of the ITO substrate was identified using a digital voltmeter based on resistance measurements.

The electrochemical deposition process was carried out using a two-electrode system, where the conductive side of the ITO glass (Indium Tin Oxide) served as the working electrode (anode), while nickel was used as the counter electrode (cathode). The electrodes were immersed in the electrolyte solution with an additional 12 mL of deionized water. A constant DC voltage of 4 V was applied for 5 minutes to form the PPy thin film.

A uniform polypyrrole thin film was deposited on the conductive surface of the ITO substrate. After deposition, the samples were rinsed with distilled water to remove residual electrolytes.

Each sample was prepared five times to ensure reproducibility.

One sample was kept as a control (without plasma treatment), while the remaining samples were exposed to cold plasma at different exposure times using a dielectric barrier discharge (DBD) system.

The DBD plasma system consists of two titanium-coated stainless-steel electrodes separated by a quartz dielectric barrier. The system operates under high-voltage alternating current (HVAC) conditions. The applied voltage was 25 kV with a frequency of 28 kHz. The distance between the upper electrode and the sample distance was 2 mm.

Cold plasma was generated by ionizing atmospheric air and applied to the samples for different exposure times (5, 10, 15, and 20 minutes). After plasma treatment, the structural properties of the samples were analyzed using X-ray diffraction (XRD), while the electrical properties were investigated using Hall effect measurements before and after treatment.

3.Results

3.1 .X-ray Diffraction (XRD)

The X-ray diffraction (XRD) analysis was performed to determine the crystallinity of the prepared samples using a Shimadzu-6000 diffractometer (Japan) with $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) operating at 30 kV. The XRD patterns of all prepared samples exhibited several characteristic diffraction peaks.

A broad diffraction peak observed for the 15 min sample centered at $2\theta = 25.1^\circ$ is attributed to the minimum interplanar spacing between the planar aromatic rings of pyrrole units, indicating the semi-crystalline nature of the PPy structure. The diffraction peaks (red mark (5 min)) at $2\theta = 29.33^\circ, 32.96^\circ, 40.9^\circ, 43.45^\circ$, and 48.01° of crystal planes (012), (003), (021), (202), and (113) correspond to crystalline potassium nitrate (KNO_3) with a rhombohedral structure, space group (R3m no. 160) with lattice parameters ($a = b = 5.2830 \text{ \AA}$, $c = 8.1460 \text{ \AA}$), ($\alpha = \beta = 90^\circ$, $\gamma = 120^\circ$), which are in good agreement with standard JCPDS data (01-081-0071).

In addition, the diffraction peaks marked in black at $2\theta = 29.63^\circ, 30.2^\circ, 34.84^\circ, 49.73^\circ$, and 59.9° correspond to the crystal planes (003), (3-11), (122), (3-14), and (125), respectively. These peaks are assigned to hexagonal In_2O_3 (ITO-related phase) with space group R-3 (No. 148), lattice parameters $a = b = 9.5590 \text{ \AA}$ and $c = 9.0370 \text{ \AA}$, and $\alpha = \beta = 90^\circ$, $\gamma = 120^\circ$, in good agreement with standard JCPDS data (98-01-2147).

The XRD results clearly demonstrate that plasma exposure leads to an increase in peak intensity, as shown in **Figure 1**. The diffraction peaks become sharper and more defined with increasing plasma treatment time, which is attributed to an increase in the crystallinity of the PPy polymer and improved molecular ordering.

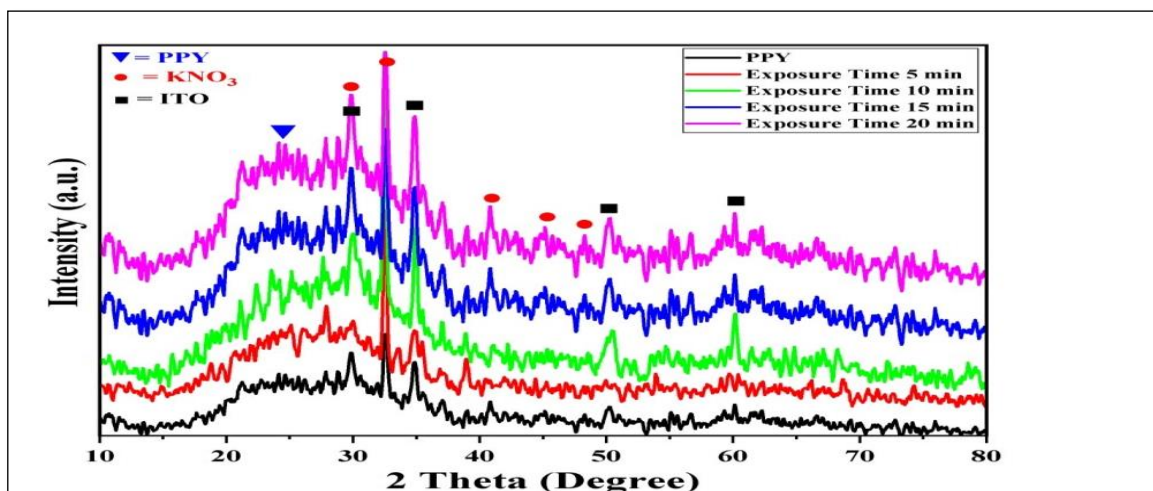


Figure 1. XRD patterns of prepared pyrrole samples deposited on ITO substrates at different exposure time of plasma.

3.2. Conductivity – Hall effect

The electrical properties of polypyrrole (PPy) thin films under dielectric barrier discharge (DBD) plasma treatment are presented in **Figures 2** and **3**. The corresponding Hall effect parameters are summarized in **Table 2**, while the electrical resistivity and conductivity values are presented in **Table 1**.

Figure 2 shows the variation of electrical resistivity (ρ) and conductivity (σ) as a function of plasma exposure time.

The untreated sample 1 (control) exhibited relatively high resistivity ($\sim 0.021 \Omega \cdot \text{cm}$) and low conductivity ($\sim 48 (\Omega \cdot \text{cm})^{-1}$). After 5 minutes (sample 2) of plasma exposure, the resistivity decreased to approximately $0.017 \Omega \cdot \text{cm}$, accompanied by an increase in conductivity to $\sim 60 (\Omega \cdot \text{cm})^{-1}$. With increasing plasma treatment time, the conductivity continued to improve. At samples 3 and 4, 10 and 15 minutes, the conductivity reached ~ 65 and $\sim 68 (\Omega \cdot \text{cm})^{-1}$, respectively. The highest conductivity ($\sim 72 (\Omega \cdot \text{cm})^{-1}$) and lowest resistivity ($\sim 0.015 \Omega \cdot \text{cm}$) were observed at sample 5) 20 minutes of plasma treatment.

Table 1. Hall effect parameters including carrier concentration (n), and mobility (μ).

Sample	Time (min)	$n (\text{cm}^{-3})$	$\mu (\text{cm}^2/\text{V}\cdot\text{s})$
1	PPY- control	4.6×10^{18}	60
2	5	1.1×10^{18}	325
3	10	4.2×10^{18}	70
4	15	4.4×10^{18}	95
5	20	2.8×10^{18}	190

Table 2. Electrical resistivity (ρ) and conductivity (σ) of Poly pyrrole (PPy) thin films.

No.	Samples	$\rho (\Omega \cdot \text{cm})$	$\sigma (\Omega \cdot \text{cm})^{-1}$
1	PPy-control	0.0208	48.07692308
2	PPy=5 min	0.0171	58.47953216
3	PPy=10 min	0.0156	64.1025641
4	PPy=15 min	0.015	66.66666667
5	PPy=20 min	0.0142	70.42253521

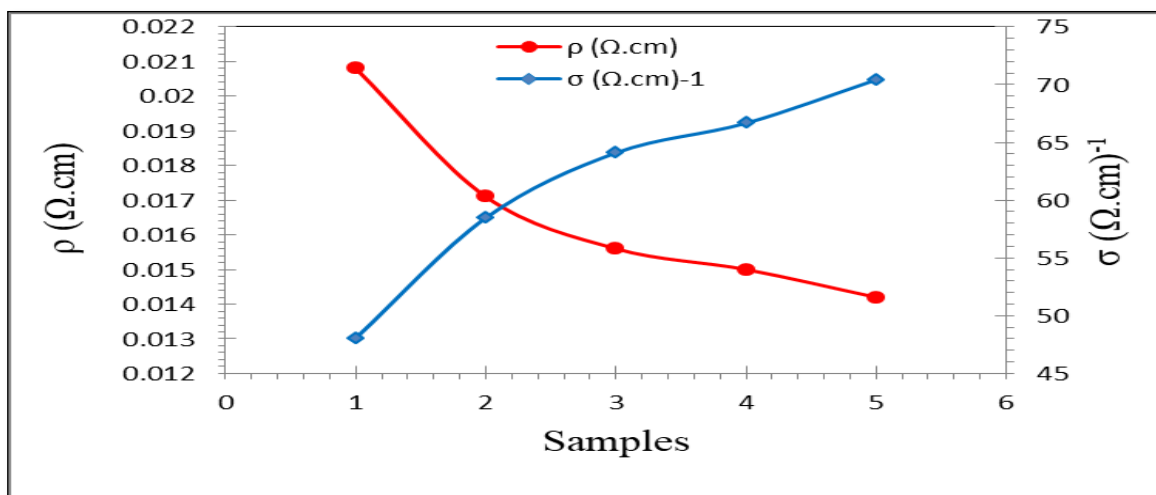


Figure 2. Resistance vs. Conductivity of PPy samples with different cold plasma exposure times: 5, 10, 15, and 20 minutes.

Figure 3 presents the variation of carrier concentration (n) and mobility (μ) with plasma exposure time. The untreated PPy film exhibited a relatively high carrier concentration ($\sim 4.6 \times 10^{18} \text{ cm}^{-3}$) and low mobility ($\sim 60 \text{ cm}^2/\text{V}\cdot\text{s}$).

After 5 minutes of plasma treatment, the carrier concentration decreased to approximately $1.1 \times 10^{18} \text{ cm}^{-3}$, while the mobility significantly increased to $\sim 325 \text{ cm}^2/\text{V}\cdot\text{s}$. With further increase in plasma exposure time, the carrier concentration increased again, while the mobility showed a decreasing trend, reaching $\sim 190 \text{ cm}^2/\text{V}\cdot\text{s}$ at 20 minutes.

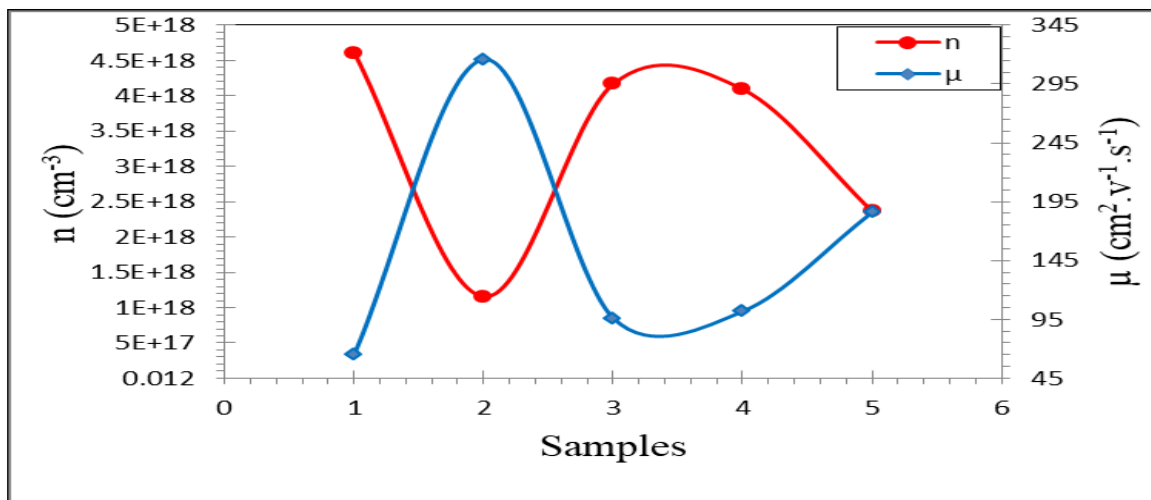


Figure 3. Mobility vs. carrier concentration of PPy samples with different cold plasma exposure times (5, 10, 15, and 20 minutes).

4. Discussion

4.1. X-ray Diffraction (XRD):

The XRD results were further analyzed and discussed in terms of structural changes induced by cold plasma treatment. The observed increase in the crystallinity of polypyrrole (PPy) films after plasma treatment can be attributed to structural rearrangement of the polymer chains, leading to improved molecular ordering. This enhancement promotes the formation of semi-crystalline regions, resulting in sharper diffraction peaks with higher intensity. In addition, plasma treatment induces partial reduction of amorphous regions within the polymer matrix, which are responsible for the broad background observed in X-ray diffraction patterns. It also reduces surface impurities that contribute to X-ray scattering, thereby improving peak definition and clarity²⁴⁻²⁷. The increase in peak intensity after plasma treatment can be attributed to structural modifications in PPy films, including improved chain alignment and enhanced inter-chain interactions. These effects are associated with the partial removal of amorphous regions, which typically contribute to the broad background in XRD patterns, as well as improved molecular stacking. In addition, plasma-induced surface activation may facilitate slight structural rearrangements, leading to increased crystalline ordering and sharper diffraction peaks²⁸⁻³¹.

4.2 Conductivity – Hall effect

The significant increase in electrical conductivity following cold plasma treatment using dielectric barrier discharge (DBD) is not merely a numerical improvement but represents a fundamental modification in the electrical transport properties of polypyrrole (PPy) thin films.

The electrical conductivity (σ) is described by the classical relation (**Equation 1**):

$$\sigma = n e \mu \quad (1)$$

where σ (S/cm) represents electrical conductivity, n (cm^{-3}) is the charge carrier concentration, e is the elementary charge ($1.602 \times 10^{-19} \text{ C}$), and μ ($\text{cm}^2/\text{V}\cdot\text{s}$) is the carrier mobility. Conductivity is also inversely related to resistivity (ρ), where $\sigma = 1/\rho$.

From this relation, it is clear that the enhanced conductivity observed in the sample treated for 20 minutes is not only due to an increase in charge-carrier concentration but also due to improved charge-transport pathways and enhanced carrier mobility induced by plasma treatment. In conducting polymers such as PPy, charge transport occurs mainly via a polaron/bipolaron hopping mechanism along conjugated polymer chains, where structural ordering and π - π

stacking significantly influence carrier mobility. This interpretation is consistent with previous studies, where structural ordering and chain organization were reported to strongly influence the electrical properties of polypyrrole systems. Furthermore, it has been shown that conductivity in PPy is highly dependent on microstructural arrangement and doping level, where improved chain alignment and reduced defect density enhance charge transport efficiency. These findings are also supported by theoretical models describing charge transport in conducting polymers as a combined effect of carrier density, conjugation, and structural ordering. However, the evolution of conductivity with plasma exposure time does not follow a strictly linear trend. The observed variations between 10 and 15 minutes indicate a competition between two simultaneous processes: plasma-induced surface activation and structural degradation.

On one hand, plasma treatment enhances carrier concentration through surface activation and oxidation processes. On the other hand, excessive ion bombardment may lead to partial polymer chain scission and increased structural disorder, which act as scattering centers that reduce carrier mobility. At a longer exposure time (20 minutes), the system reaches a quasi-stable state where the increase in carrier concentration and improved network connectivity compensate for defect-induced scattering, resulting in the maximum observed conductivity. To further validate the obtained results, A comparison was made between the electrical conductivity obtained in the present work and the previously reported studies on PPy thin films. In this study, the highest conductivity was obtained to be about $72 (\Omega \cdot \text{cm})^{-1}$ after 20 min of DBD plasma treatment. The conductivity of this film is similar to, or better than, many reported values using chemical methods in the literature, which yielded conductivities ranging from $30\text{--}80 (\Omega \cdot \text{cm})^{-1}$ with the addition of surfactants, and $10\text{--}55 (\Omega \cdot \text{cm})^{-1}$ for chemically synthesized PPy films. The results show that the DBD plasma treatment is effective method to increase the electrical conductivity of PPy thin films³²⁻³³.

5. Conclusion

After exposure to cold atmospheric plasma (CAP), the structural and electrical properties of polypyrrole (PPy) thin films were significantly improved. The X-ray diffraction results revealed an increase in peak intensity and sharper diffraction features, particularly at longer exposure times (up to 20 minutes), indicating an enhancement in the crystallinity of the PPy polymer.

This improvement can be attributed to plasma-induced structural modifications, including partial removal of amorphous regions and surface impurities, which typically contribute to background scattering in XRD patterns. In addition, plasma treatment promotes molecular reorganization, improved chain alignment, and enhanced inter-chain interactions, leading to a more ordered polymer structure.

Electrical measurements based on the Hall effect demonstrated a clear dependence on plasma exposure time (5, 10, 15, and 20 minutes). The untreated PPy film exhibited high electrical resistance and low conductivity, indicating limited charge carrier transport. In contrast, plasma-treated samples showed a significant reduction in resistance and an increase in conductivity, confirming improved charge transport properties.

The enhancement in electrical performance is attributed to plasma-induced surface activation, which facilitates increased carrier mobility and improved electrical pathways within the polymer matrix. However, the results also suggest that the plasma effect is time-dependent, where both beneficial modifications and possible structural disturbances may occur depending on exposure duration.

Overall, these findings confirm the effectiveness of cold plasma treatment as a controllable and efficient method for tailoring the structural and electrical properties of conducting polymers such as PPy. Future work is recommended to investigate different plasma sources, a wider range of treatment conditions, and potential practical applications in electronic and sensing devices, which would further expand the understanding and applicability of plasma-modified polymer systems.

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Conflict of Interest

The authors declare that there is no conflict of interest.

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Ethical Clearance

The present study did not involve any experiments on human participants or animal subjects; therefore, ethical approval was not required. All experiments complied with the ethical and safety policies of our institution and the national research regulations of Iraq.

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