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# SYNTHESIS AND CHARACTERIZATION OF CHITOSAN BASED POLYPYRROLE COPOLYMER AND EVALUATION OF ITS OPTICAL PROPERTIES

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**Abstract**
Biopolymer-based conducting materials have emerged as attractive candidates for sustainable electronic applications. In this study, a chitosan–polypyrrole (CS–PPy) copolymer was synthesized through in situ chemical oxidative polymerization and characterized using FTIR, solid-state NMR, XRD, SEM–EDS and UV–visible spectroscopy. Structural analyses confirmed successful copolymer formation while XRD revealed a predominantly amorphous nature with reduced crystallinity. SEM images showed a rough- granular morphology with uniform polypyrrole distribution and EDS verified the presence of C, N, O and S elements. UV–visible studies demonstrated enhanced optical absorption and the optical band gap was determined to be 2.09 eV using Tauc's method. The improved structural and optical properties indicate that the synthesized CS–PPy copolymer is a promising sustainable conducting material for applications in optoelectronics, sensors and advanced functional devices.

**Keywords:** Chitosan, Polypyrrole, Conducting polymer, Optical properties, Biopolymer composite

## 1. Introduction

The development of advanced functional materials with tailored electrical, optical, and physicochemical properties has become an important area of research due to their potential applications in sensors, energy storage systems, optoelectronic devices, and environmental technologies [1]. Among intrinsically conducting polymers, polypyrrole (PPy) has received considerable attention because of its high electrical conductivity, environmental stability, facile synthesis, and versatile functional characteristics. These properties make PPy a promising candidate for numerous technological applications [2]. However, its practical implementation is often limited by poor mechanical strength, limited processability, and insufficient structural flexibility. To address these shortcomings, researchers have increasingly focused on the design of hybrid materials and copolymers incorporating natural polymers with conducting polymer networks [3]. Chitosan, a naturally derived polysaccharide obtained through the deacetylation of chitin, has emerged as an attractive biopolymer for the fabrication of multifunctional composite materials. Owing to its biodegradability, biocompatibility, non-toxicity, and abundance of amino and hydroxyl functional groups, chitosan provides an excellent platform for chemical modification and composite formation [4]. The presence of these reactive groups facilitates strong

intermolecular interactions with conducting polymers, resulting in improved structural integrity and enhanced material performance. Moreover, the utilization of chitosan aligns with the growing demand for environmentally sustainable materials in advanced technological applications [5]. The combination of chitosan and polypyrrole offers a promising approach for developing hybrid materials with synergistic properties. In such systems, chitosan contributes mechanical stability, flexibility, and processability, whereas polypyrrole provides electrical conductivity and electronic functionality [6]. The interactions between the polymer chains can significantly influence the structural arrangement and electronic behavior of the resulting copolymer. Consequently, understanding these interactions is essential for optimizing the performance of the material for specific applications. Optical properties represent a key aspect in the characterization of conducting polymer-based materials because they provide valuable insights into electronic transitions, charge transport mechanisms, and energy band structures [7]. Parameters such as absorbance, optical band gap, and absorption coefficient are closely related to the molecular architecture and degree of conjugation within the polymer matrix. Therefore, the investigation of optical characteristics is crucial for assessing the suitability of these materials for optoelectronic and photonic applications [8].

In the present study, a chitosan-based polypyrrole copolymer was synthesized and systematically characterized to evaluate its structural and optical properties. Particular emphasis was placed on examining the optical behavior of the synthesized material and elucidating the relationship between copolymer formation and its electronic characteristics. The findings are expected to contribute to the development of sustainable conducting polymer systems with enhanced functionality for future technological applications [9].

## 2. Materials and Methods

### *2.1 Materials*

Chitosan from Shrimp Shells (Mol. Wt.: 3800-20000, Degree of Deacetylation: >=75.00%) (HIMEDIA) was used as the base polymer. Pyrrole reagent grade (98%) (SIGMA-ALDRICH), Ammonium persulfate (APS) was used as the oxidizing agent. Acetic acid, Deionized water and ethanol were of analytical grade and used without further purification.

### *2.2 Synthesis of Chitosan–Polypyrrole (CS–PPy) Copolymer*

The CS–PPy copolymer was synthesized via *in-situ* chemical oxidative polymerization of pyrrole in a chitosan matrix (Figure 1). Chitosan (1 g) was dissolved in 100 mL of 1% (v/v) aqueous acetic acid under continuous magnetic stirring at room temperature (≈25 °C) for 4–6 h to obtain a homogeneous solution which was filtered to remove undissolved impurities. Pyrrole monomer (0.5–1 mL) was added dropwise to the chitosan solution under constant stirring and allowed to disperse for 30 min. An oxidizing solution was prepared by dissolving 1.5 g of ammonium persulfate in 50 mL of deionized water. The reaction mixture was maintained at 0–5 °C using an ice bath and the oxidant solution was added dropwise over 30–60 min under constant stirring using Magnetic stirrer. The mixture was stirred using magnetic stirrer at 0–5 °C for 4 h and subsequently at room temperature for 12–24 h to ensure complete polymerization. The resulting black precipitate was filtered through Whatman filter paper No. 40, washed repeatedly with distilled water followed by ethanol to remove residual reactants and dried in an oven at 50 °C for 10 h [10].

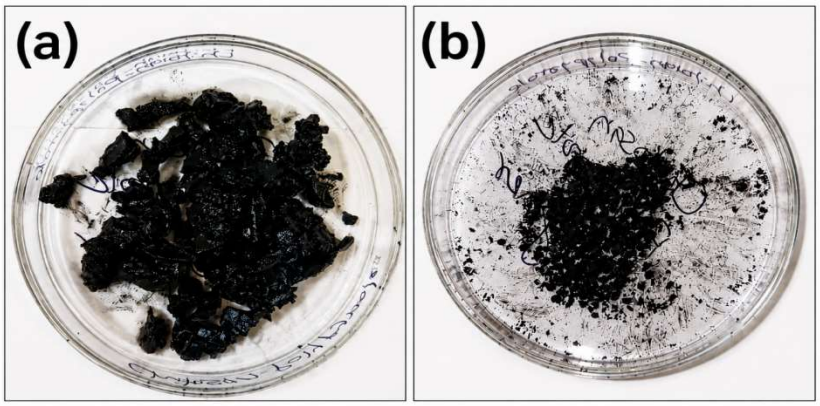



**Figure 1. (a)** Black precipitate obtained after *in-situ* oxidative polymerization of pyrrole in the chitosan matrix, **(b)** synthesized CS–PPy copolymer after drying at **50 °C for 10 h.**

### *2.3 Optical properties measurement*

The optical properties of the synthesized chitosan-based polypyrrole copolymer were investigated using ultraviolet–visible (UV–Vis) spectroscopy. The absorption spectra were recorded employing a UV–Vis spectrophotometer Shimadzu UV-VIS-IR (Model: UV: 3600 Plus) over a wavelength range of 200-800 nm at ambient temperature. The prepared copolymer samples were subjected to spectroscopic examination to evaluate their light absorption behavior and electronic transition characteristics. The UV–Vis absorption spectra were acquired and analyzed to determine the optical response of the material within the ultraviolet and visible regions. The observed absorption features were further utilized to assess the influence of copolymer formation on the electronic structure of the synthesized material [11].

### 2.4 Characterization Techniques

The synthesized chitosan–polypyrrole (CS–PPy) copolymer was characterized using various analytical techniques. Solid-state $^{13}C$ NMR spectroscopy was employed to investigate the chemical environment of carbon atoms and to examine the interactions between chitosan and polypyrrole chains. FTIR spectroscopy was used to identify the characteristic functional groups and confirm successful copolymer formation. Structural changes and crystallinity variations resulting from copolymerization were evaluated by X-ray diffraction (XRD) analysis. Surface morphology and elemental composition were examined using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS) [12].

## 3. Results and Discussion

### *3.1 FT-IR Analysis*

The FTIR spectrum of the CS–PPy copolymer (Figure 2) exhibited characteristic absorption bands of both chitosan and polypyrrole, confirming successful copolymer formation. The band at 1539 $cm^{-1}$ was attributed to the C=C stretching vibration of the pyrrole ring, indicating the presence of the polypyrrole backbone. The peak observed at 1032 $cm^{-1}$ corresponds to the C–O stretching vibration of chitosan. Weak bands appearing at 2322, 2175, 2108, 1988 and 1939 $cm^{-1}$ are associated with overtone and combination vibrations. The coexistence of these characteristic bands suggests effective intermolecular interactions between chitosan and polypyrrole chains resulting in the formation of a stable CS–PPy copolymer structure [13].

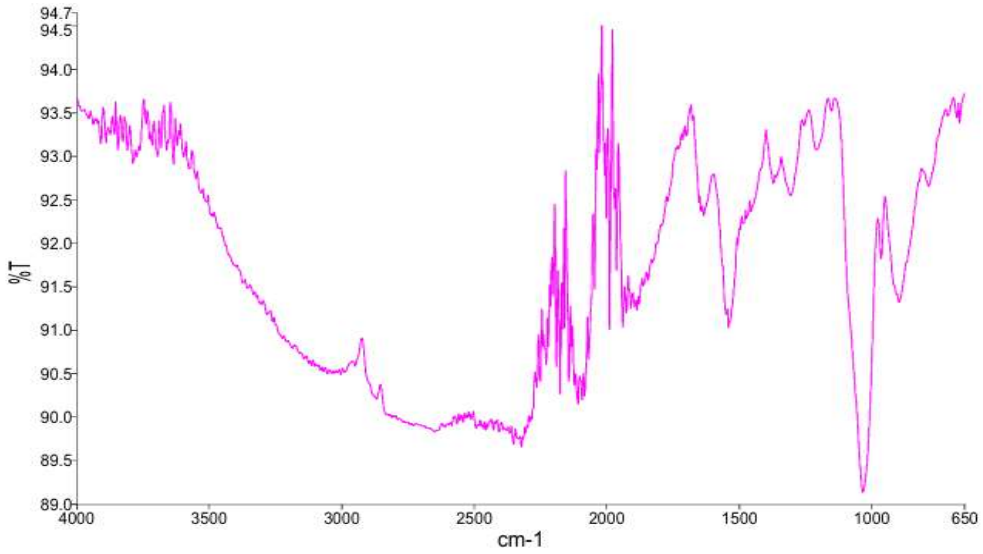



**Figure 2.** FTIR spectrum of chitosan-based polypyrrole (CS–PPy) copolymer

### *3.2 XRD Analysis*

The X-ray diffraction profile of the CS–PPy copolymer (Figure 3) displayed a broad diffraction band centered at 2θ = 22.87° corresponding to an interplanar spacing (d-value) of approximately 3.88 Å. The broad nature of this reflection and the absence of distinct, intense diffraction peaks suggest that the synthesized material possesses a predominantly amorphous character with limited semi-crystalline domains. This structural behavior indicates a significant reduction in the ordered arrangement of chitosan chains following copolymer formation. The incorporation and in-situ polymerization of polypyrrole within the chitosan matrix likely disrupt the native hydrogen-bonding network and regular chain packing of chitosan. Such a decrease in structural order is commonly observed in chitosan-based conducting polymer systems and is considered indicative of successful integration of polypyrrole into the biopolymeric framework [14].

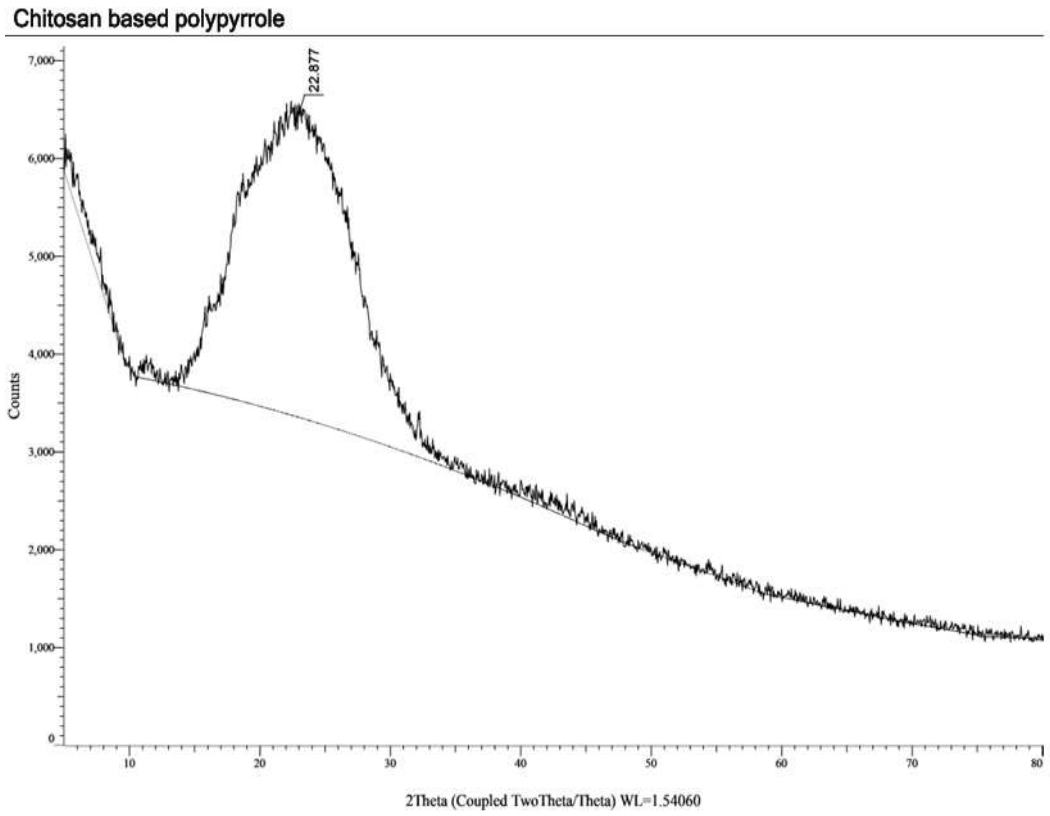



**Figure 3.** X-ray diffraction (XRD) pattern of chitosan-based polypyrrole (CS–PPy) copolymer

### *3.3 SEM Analysis*

The surface morphology of the synthesized CS–PPy copolymer was examined using SEM, and the corresponding micrographs are presented in Figure 4. At lower magnifications- the material exhibits a compact and continuous surface with a heterogeneous texture indicating the successful incorporation of polypyrrole into the chitosan matrix. Higher-magnification images reveal a granular, cauliflower-like morphology characteristic of polypyrrole suggesting its uniform distribution throughout the polymer network. The surface consists of interconnected nodular structures with noticeable roughness reflecting effective polymer growth during the in-situ oxidative polymerization process. The interconnected and porous morphology is expected to facilitate charge transport and ion diffusion, supporting the semiconducting behavior observed in optical studies. The rough surface provides an increased active surface area making the material suitable for applications in biosensors, supercapacitors, and optoelectronic devices. The SEM analysis confirms the successful formation of a structurally integrated CS–PPy copolymer with favorable morphological characteristics [15].

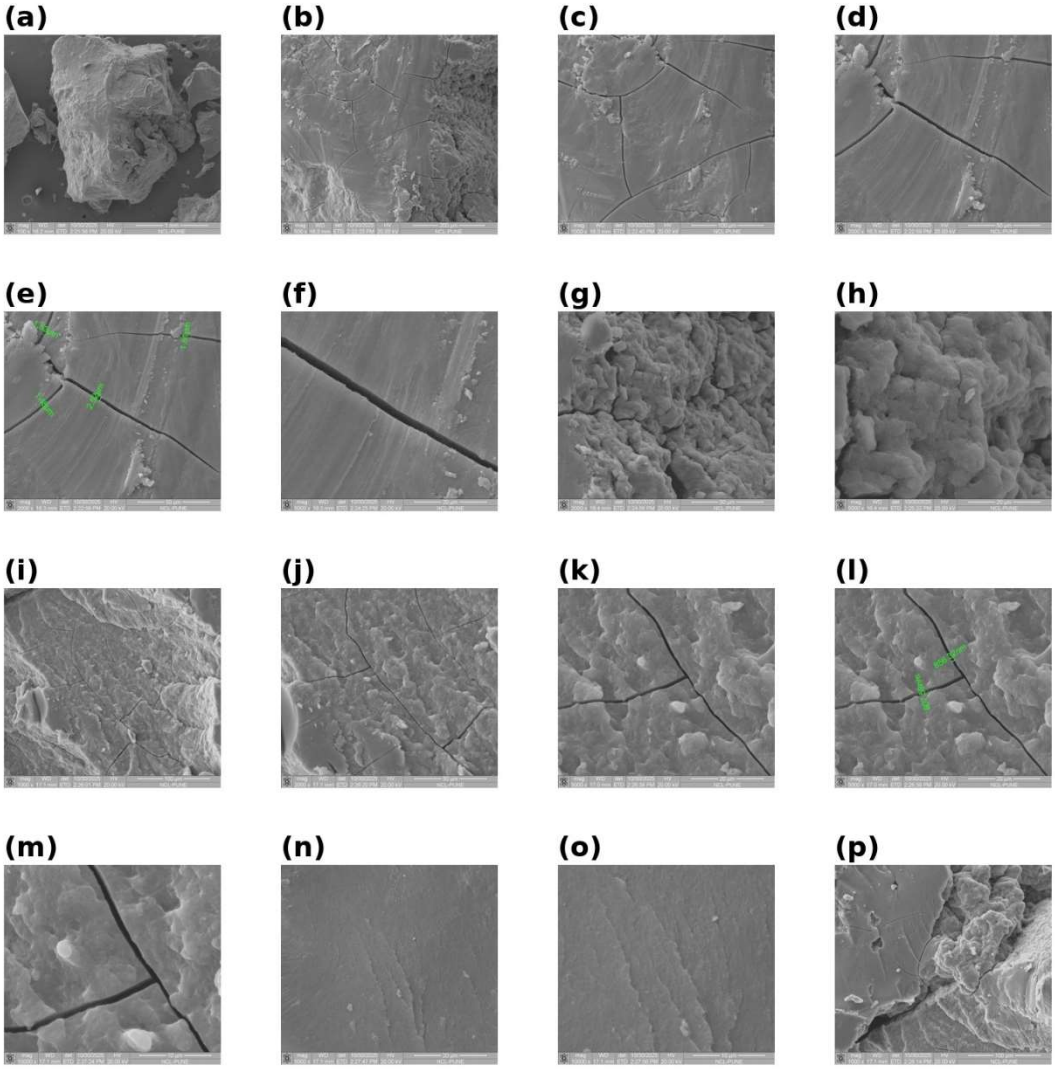
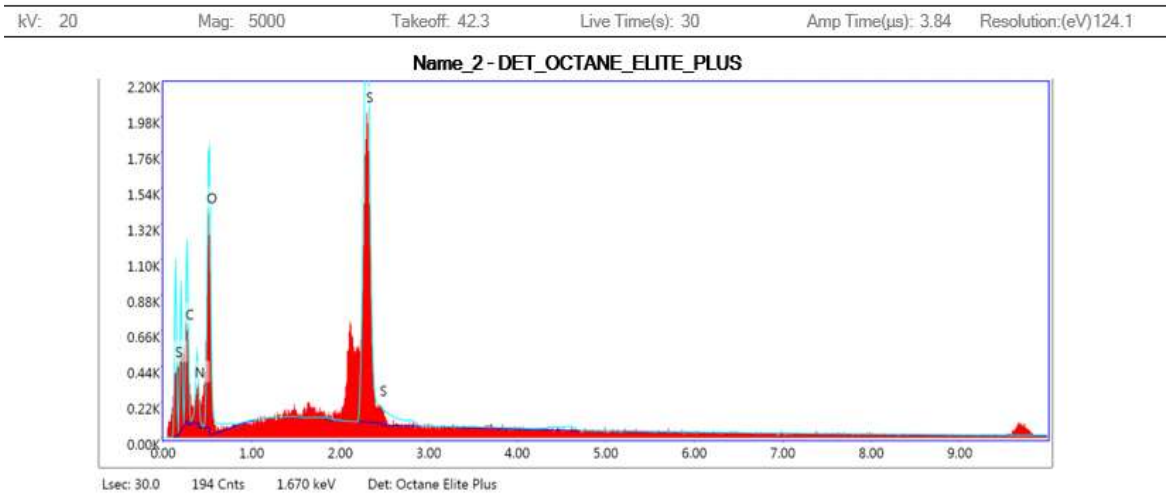



**Figure 4.** SEM micrographs of chitosan-based polypyrrole (CS–PPy) copolymer at different regions and magnifications

### *3.4 EDS Analysis*

The elemental composition of the synthesized CS–PPy copolymer was examined by EDS analysis and the corresponding spectrum is shown in Figure 5. The spectrum confirms the presence of carbon (C), nitrogen (N), oxygen (O) and sulfur (S) as the major constituent elements of the copolymer. The prominent carbon and nitrogen peaks are attributed to the polypyrrole backbone while the oxygen signal originates from the hydroxyl and other oxygen-containing functional groups of chitosan. The detection of sulfur is associated with sulfate counter ions derived from ammonium per sulfate used during oxidative polymerization indicating the successful doping of polypyrrole chains. The absence of significant impurity peaks suggests the high purity of the synthesized material. The EDS results verify the successful formation of the CS–PPy copolymer and support the effective incorporation of both chitosan and polypyrrole within the polymeric network [16].




**eZAF Smart Quant Results**

| Element | Weight % | Atomic % | Net Int. | Error % | Kratio | Z | A | F |
|---|---|---|---|---|---|---|---|---|
| C K | 32.38 | 41.63 | 144.42 | 11.16 | 0.0652 | 1.0450 | 0.1926 | 1.0000 |
| N K | 14.51 | 16.00 | 58.94 | 14.94 | 0.0183 | 1.0207 | 0.1236 | 1.0000 |
| O K | 34.71 | 33.51 | 296.58 | 10.99 | 0.0572 | 0.9996 | 0.1648 | 1.0000 |
| S K | 18.40 | 8.86 | 833.16 | 2.56 | 0.1595 | 0.8889 | 0.9714 | 1.0033 |

**Figure 5.** Energy-dispersive X-ray spectroscopy (EDS) spectrum of chitosan-based polypyrrole (CS–PPy)

### *3.5 UV–Visible and Optical Properties*

The UV–visible absorption spectrum of the CS–PPy copolymer (Figure 6) exhibited broad absorption across both the ultraviolet and visible regions indicating enhanced optical activity. The increased absorption in the visible region can be attributed to π–π* electronic transitions within the conjugated polypyrrole chains incorporated into the chitosan matrix. The optical band gap was determined using the Tauc method assuming a direct allowed transition. As shown in Figure 7, extrapolation of the linear portion of the $(\alpha h\nu)^2$ versus hν plot yielded a band gap value of 2.09 eV. This result confirms the semiconducting nature of the synthesized copolymer and falls within the typical range reported for polypyrrole-based conducting materials. The observed band gap suggests that the conjugated electronic structure of polypyrrole is largely preserved after copolymerization with chitosan. Minor variations in band gap energy may be associated with structural disorder, intermolecular interactions, and changes in polymer chain arrangement. The moderate band gap value indicates improved charge delocalization and enhanced electronic communication within the copolymer network making the material promising for applications in optoelectronics, biosensing, and energy-storage devices. The UV–Vis analysis confirms the successful formation of a semiconducting CS–PPy copolymer with favorable optical properties and a well-defined direct electronic transition [17].

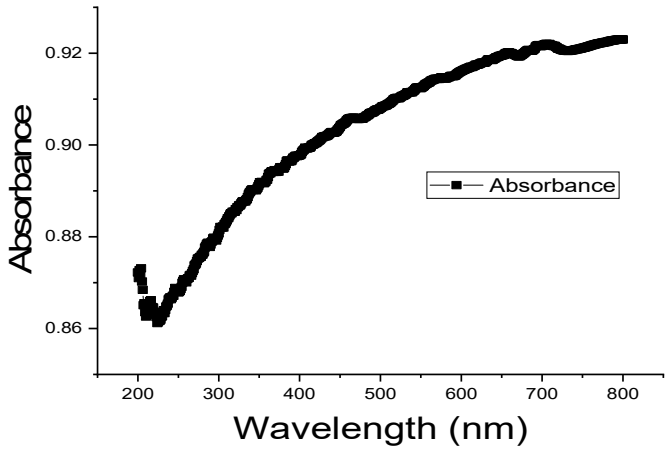



**Figure 6.** UV–Visible absorption spectrum of chitosan-based polypyrrole (CS–PPy) copolymer

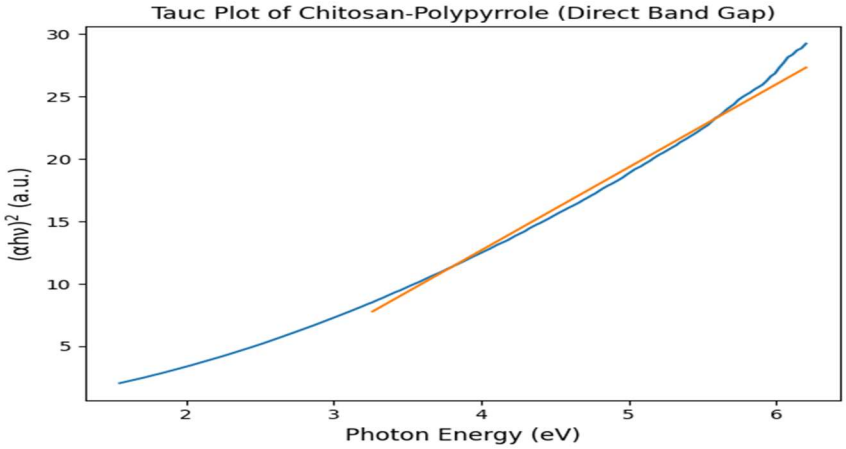



**Figure 7.** Tauc plot of chitosan–polypyrrole (CS–PPy) copolymer derived from UV–Vis absorption spectra

### *2.1.1 3.6 Solid-State NMR Analysis*

The solid-state $^{13}C$ NMR spectrum of the CS–PPy copolymer (Figure 8) displays characteristic signals arising from both chitosan and polypyrrole, confirming successful copolymer formation. The resonance at 175.6 ppm is assigned to the carbonyl carbon of residual acetyl groups in chitosan while the broad peak at 127.8 ppm corresponds to the conjugated carbon atoms of the polypyrrole backbone. Signals observed at 100.5, 75.7, 72.4 and 57.4 ppm are attributed to the glucosamine ring carbons of chitosan. Additional resonances between 24.0 and 36.4 ppm arise from aliphatic carbon environments within the copolymer matrix. The presence of characteristic carbon resonances from both components confirms the successful incorporation of polypyrrole into the chitosan framework. [18].

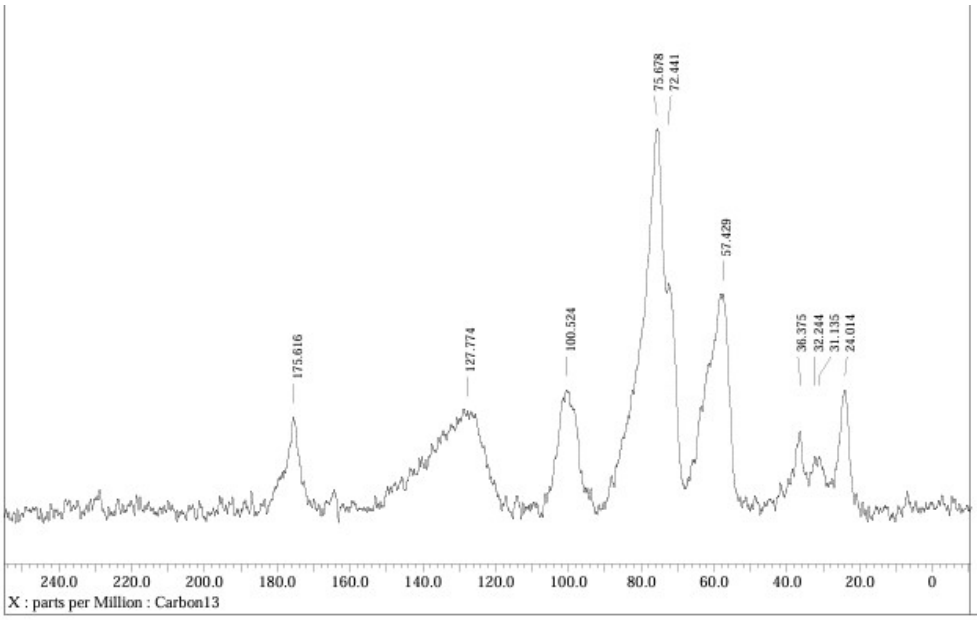



**Figure 8.** Solid-state $^{13}C$ NMR spectrum of chitosan-based polypyrrole (CS–PPy) copolymer

### *3.7 Optical Properties*

The absorbance values (0.86119–0.92300 AU) indicate that the CS–PPy copolymer exhibits consistently high optical absorption throughout the investigated spectral region (Table 1). The small variation in absorbance suggests a homogeneous film with nearly uniform optical behaviour. The corresponding transmittance values (11.94–13.77 %) demonstrate that only a limited fraction of the incident radiation passes through the polymer film confirming its excellent light attenuation capability. Using the measured film thickness of 50 μm, the absorption coefficient was calculated to vary between 396.66 and 425.13 $cm^{-1}$. These values indicate efficient photon absorption by the conjugated polymeric network and confirm the semiconducting nature of the synthesized material. The extinction coefficient ranged from $6.39 \times 10^{-4}$ to $2.71 \times 10^{-3}$, reflecting moderate optical attenuation within the visible region. The increase in *k* toward shorter wavelengths is attributed to stronger electronic transitions associated with the π-conjugated backbone of polypyrrole (Table 1).

**Table 1.** Optical Properties of CS–PPy copolymer

| Sr. No. | Optical Properties | Calculated value from UV-Vis Data |
|---|---|---|
| 1 | Absorbance **(A)** | 0.86119–0.92300 |
| 2 | Transmittance **(T)** | 11.94–13.77 % |
| 3 | Absorption coefficient **(α)** | 396.66–425.13 $cm^{-1}$ |
| 4 | Extinction coefficient **(k)** | $(6.39 \times 10^{-4}) – (2.71 \times 10^{-3})$ |
| 5 | Optical band gap **(Eg)** | 2.09 eV |

## 4. Conclusion

The chitosan–polypyrrole (CS–PPy) copolymer was successfully prepared through an in-situ chemical oxidative polymerization approach. Structural characterization by FTIR and solid-state NMR verified the formation of the copolymer through the presence of characteristic functional groups and carbon environments. XRD analysis indicated a largely amorphous nature with reduced crystallinity, while SEM examination revealed a rough, granular surface morphology with well-dispersed polypyrrole domains. The elemental composition determined by EDS confirmed the incorporation of carbon, nitrogen, oxygen, and sulfur within the copolymeric framework. Furthermore, UV–visible studies demonstrated enhanced light absorption along with a reduced optical band gap, highlighting the improved optical performance of the material. Collectively, these findings suggest that the synthesized CS–PPy copolymer possesses desirable structural and optical characteristics, making it a promising material for applications in optoelectronics, sensing technologies, and other advanced functional polymer systems.

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