

Exploring Synthesis, Characterization, and Applications of Conducting Polypyrrole and Metal Oxide Composites: A Multifaceted Investigation

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Abstract- This research aims to investigate the synthesis, characterization, and potential applications of conducting polypyrrole and its composites, with a specific focus on metal oxide combinations. The primary objective is to understand the intricate structural features of these materials and explore their applicability in diverse technological domains. The methodology involves a systematic approach, commencing with the synthesis of polypyrrole, metal oxides, and their composites through established methods. Material selection is crucial, considering the delicate balance between polypyrrole's inherent electrical conductivity and the desirable properties of composite materials. Subsequently, the materials undergo comprehensive characterization using various analytical techniques such as FTIR spectroscopy, UV spectroscopy, XRD, TEM, and EIS, providing detailed insights into their molecular and structural attributes. The findings reveal a nuanced understanding of the synthesized materials. FTIR spectroscopy exposes distinctive chemical bonds and functional groups, UV spectroscopy highlights electronic transitions, XRD unveils crystalline structures, TEM provides high-resolution images of nanoparticle morphology, and EIS assesses electrical properties. The data collectively contributes to a comprehensive comprehension of the materials' characteristics. This research not only establishes a solid foundation for the tailored synthesis and characterization of conducting polypyrrole and its composites but also explores their potential applications in sensors, energy storage devices, and electronic systems. The holistic approach underscores the transformative potential of these materials across diverse technological frontiers.

Keywords- Polypyrrole, Characterisation, Electrochemical, polymerization, transmission electron microscopy (TEM)

1. Introduction

Conducting polypyrrole (PPy) is a prominent area of research in the field of conducting polymers, owing to its unique electronic and electrochemical properties. PPy is a conjugated polymer that exhibits electrical conductivity, making it suitable for various applications, including sensors, actuators, and electronic devices. The synthesis of PPy involves the chemical polymerization of pyrrole monomers, typically initiated by an oxidizing agent. The polymerization process leads to the formation of a conductive backbone, composed of alternating single and double bonds. Doping PPy with various dopants further enhances its conductivity and performance in specific applications (Raicopol et al., 2013; Zhang et al., 2013). One notable characteristic of PPy is its redox activity, allowing it to undergo reversible doping and dedoping processes. This feature is exploited in applications such as batteries and supercapacitors, where PPy serves as an electrode material. The electrochemical behavior of PPy is influenced by factors like dopant type, synthesis method, and film morphology. In addition to its electrical conductivity, PPy possesses good environmental stability and mechanical flexibility, making it an attractive candidate for flexible electronics. Researchers have explored different strategies to improve the mechanical properties

of PPy, such as compositing with other materials or modifying the synthesis conditions. The composite materials involving PPy have gained significant attention due to their synergistic properties. Incorporating PPy into composites with various substances, such as carbon-based materials (carbon nanotubes, graphene), metal oxides, or polymers, can impart additional functionalities to the resulting materials. For instance, PPy/carbon composites exhibit enhanced electrical conductivity and mechanical strength, making them suitable for applications in energy storage devices and sensors. The application of PPy-based materials extends to sensing technologies. PPy's conductivity changes in response to external stimuli, enabling its use in sensors for detecting gases, humidity, and biomolecules. Functionalizing PPy or incorporating it into composite structures enhances its sensing performance and selectivity. The versatility of PPy has led to its exploration in biomedical applications. PPy and PPy-based composites have shown promise in drug delivery systems, bioelectrodes, and tissue engineering (Abbasi, Militky, et al., 2013; Ofek Almog et al., 2012; Ohtsuka, 2012). The biocompatibility and electroactivity of PPy make it an intriguing material for interfacing with biological systems.

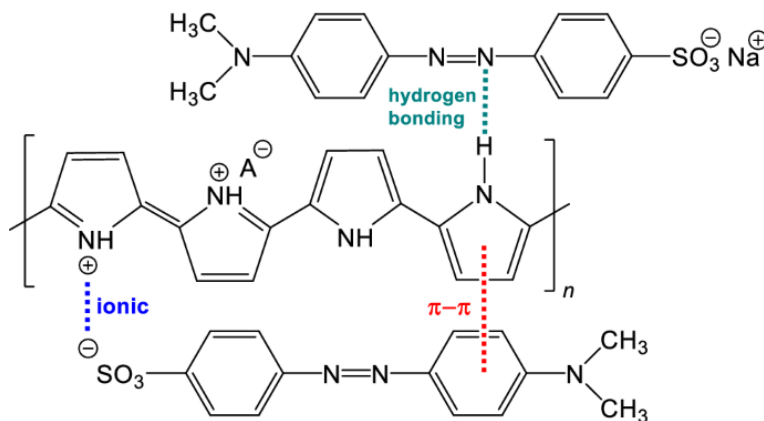


Figure 1 Conducting polypyrrole (PPy)

Despite the remarkable progress in the field of conducting polypyrrole, challenges remain. Achieving better control over the synthesis process to obtain reproducible properties, optimizing the performance of PPy in specific applications, and understanding its long-term stability are ongoing research focuses, conducting polypyrrole and its composites represent a dynamic area of research with wide-ranging applications. The unique electrical, electrochemical, and mechanical properties of PPy, along with the synergistic effects achieved through compositing, make it a material of great interest for advancements in electronics, sensors, energy storage, and biomedical applications. Ongoing research continues to unravel the full potential of PPy and its composites, paving the way for innovative technologies in various fields. Conducting polypyrrole (PPy) and its composites stand at the forefront of advanced materials research due to their unique properties and versatile applications. PPy is a conjugated polymer known for its remarkable electrical conductivity, making it a pivotal element in the development of various technologies. The synthesis of PPy involves the chemical polymerization of pyrrole monomers, often initiated by an oxidizing agent (Ilicheva et al., 2012; Janmanee et al., 2012; Keothongkham et al., 2012). This process forms a conductive polymer backbone with alternating single and double bonds. Doping PPy with various substances further enhances its conductivity, allowing for tailored applications in diverse fields. One of PPy's distinguishing features is its redox activity, facilitating reversible doping and dedoping processes. This characteristic finds application in energy storage devices like batteries and supercapacitors, where PPy serves as an electrode material. The electrochemical

behavior of PPy is influenced by parameters such as the type of dopant, synthesis method, and film morphology. Beyond its electrical properties, PPy exhibits notable environmental stability and mechanical flexibility, making it an attractive candidate for flexible electronics. Researchers have explored strategies to improve PPy's mechanical properties, including composite materials and modified synthesis conditions. Composites involving PPy have garnered significant attention due to the synergistic effects achieved by combining it with other materials. Incorporating PPy into composites with substances like carbon nanotubes, graphene, metal oxides, or polymers enhances the resulting materials' functionalities. PPy/carbon composites, for instance, exhibit improved electrical conductivity and mechanical strength, making them suitable for applications in energy storage and sensors. The versatility of PPy extends to sensing technologies, where its conductivity changes in response to external stimuli. PPy-based sensors find applications in detecting gases, humidity, and biomolecules. Functionalizing PPy or incorporating it into composite structures enhances its sensing performance and selectivity. Moreover, PPy and its composites have shown promise in biomedical applications. Their biocompatibility and electroactivity make them suitable for drug delivery systems, bioelectrodes, and tissue engineering (Ansari, 2006; Saoudi et al., 2004; Zerbino et al., 2011). These applications underscore the potential of PPy in interfacing with biological systems for therapeutic purposes. Despite these advancements, challenges persist in the field of conducting polypyrrole. Achieving precise control over synthesis processes for reproducibility, optimizing PPy's performance in specific applications, and understanding its long-term stability are ongoing areas of research focus.

In the process of synthesising polypyrrole, electrochemical polymerization provides an alternate method that is also an extremely adaptable one. It is possible to polymerize pyrrole monomers on a conductive substrate by employing this technique, which involves electrochemical oxidation. The voltage of the electrode is responsible for controlling the beginning and development of the polymer chain, which in turn provides a significant amount of control over the thickness and morphology of the film. The process of electrochemical polymerization enables the fine tailoring of polypyrrole characteristics by the modification of factors like as the concentration of the monomer, the potential of the electrode, and the composition of the electrolyte component. Because of this level of control, it is especially suitable for applications in which particular material qualities are of utmost importance. When it comes to understanding the qualities and performance of polypyrrole, structural characterisation is an extremely important factor. The structural characteristics of the material are deciphered by the application of a variety of methods, each of which provides a distinct perspective. Spectroscopic methods, such as Fourier-transform infrared (FTIR) spectroscopy and Raman spectroscopy, are extremely important in the process of elucidating the structural particulars of polypyrrole. Information on the functional groups that are present in the polymer can be obtained by FTIR, which provides insights into the chemical makeup of the material. Raman spectroscopy, on the other hand, is able to disclose vibrational modes and can be utilized to evaluate the degree of conjugation that exists within the polymer backbone. When it comes to confirming the effective synthesis of polypyrrole, detecting functional groups, and gaining a knowledge of the molecular arrangement inside the polymer structure, these spectroscopic approaches serve as powerful instruments. There is information that may be obtained regarding the crystallinity, morphology, and molecular arrangement of polypyrrole by the utilization of X-ray and neutron scattering techniques. These techniques include X-ray diffraction (XRD) and small-angle neutron scattering (SANS). X-ray diffraction (XRD) is a very helpful technique for determining the crystalline character of polypyrrole films, whereas scanning electron microscopy

(SANS) provides insights into the shape and arrangement of polymer chains at the nanoscale. The use of these scattering techniques contributes to a more in-depth understanding of the structural properties of the material that influence its performance in a variety of applications. Researchers have the ability to see the surface morphology and internal structure of polypyrrole through the use of morphological analysis techniques such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM). SEM is able to produce high-resolution images of the surface, but transmission electron microscopy (TEM) may provide precise insights into the internal structure and shape of nanoparticles. When it comes to adapting the properties of polypyrrole to specific applications, such as sensors, conductive coatings, or energy storage devices, having a solid understanding of the morphology of polypyrrole is absolutely necessary. Information regarding the electrochemical behavior of polypyrrole and its capacity to store charge can be obtained through the use of electrochemical techniques such as cyclic voltammetry and impedance spectroscopy.

2. Literature review

Anh 2021 et. al was fabricated onto a microelectrode for cholesterol sensing application. The cholesterol oxidase (ChOx) was immobilized on the CeO₂ NPs/PPy/electrode by the physical adsorption route. The structure and morphology of the CeO₂ NPs/PPy nanocomposite were characterized by X-ray diffraction, field emission scanning electron microscopy, and energy dispersive X-ray spectroscopy. Results showed that the ChOx/CeO₂ NPs/PPy/electrode was linearly related with cholesterol in the range of 50 to 500 mg/dL. The sensitivity of ChOx/CeO₂ NPs/PPy/electrode was 5.7×10^{-6} mA/mg·dL⁻¹. The optimal parameters, including pH value and temperature, and selectivity, storage stability, and reproducibility of ChOx/CeO₂ NPs/PPy/electrode were investigated (Anh et al., 2021).

Li 2019 et. al were successfully prepared via the solution chemistry method using pyrrole (Py) as raw material, ammonium persulfate (APS) as oxidant, and cetyltrimethyl ammonium bromide (CTAB) as surfactant. The ZnO/PNR composite was synthesized with zinc oxide nanoparticles absorbed on the surface of PPy nanorings through the one-pot in situ sol-gel method. The composite shows a three-dimensional intertwined network structure where the size of polypyrrole nanorings ranges from 80 nm to 100 nm in diameter and the average size of uniformly distributed ZnO nanocrystals is 10.49 nm. The unique three-dimensional conductive framework can provide good electronic contact between the ZnO particles and buffer the volume variation during the lithiation/delithiation processes. As an electrode material for LIBs, the ZnO/PNR composite delivers a first cycle discharge capacity of 1658 mAh g⁻¹ and a capacity retention of 50.7% over 150 cycles at 200 mA g⁻¹, indicating high specific capacity and outstanding cycle stability (Li et al., 2019).

Tikish 2018 et. al were prepared by solution processing method, using dimethyl sulfoxide (DMSO) solvent. Characterization of the polymer blends was carried out based on the data analysis from Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and differential scanning calorimetry (DSC). The PPy/PANI system was successfully formed blends in DMSO solvent. The polymer blends showed almost amorphous nature in XRD spectra because of intermolecular interaction between PPy and PANI macromolecules, which was confirmed by FT-IR data. Specifically, the DSC result for the PPy: PANI = 50: 50 wt.% blend showed only one

glass transition temperature (T_g), which indicates that the two polymers are well miscible without undergoing any phase separation (Tikish et al., 2018).

Steffens 2018 et. al was investigated under the influence of relative humidity. The variations in the deflection of the coated cantilevers when exposed to relative humidity were evaluated. The results indicated a linear sensitivity in ranges, where the high value was obtained for a polypyrrole-sensitive layer between 20 and 45% of humidity. Furthermore, the sensor shows excellent performance along with rapid response and recovery times, relatively low hysteresis, and excellent stability. The sensors developed are potentially excellent materials for sensing low humidity for long time (Steffens et al., 2018).

Maity 2015 et. al prepared by in situ chemical polymerization of pyrrole using suitable oxidant and dopant. These composite fabrics show surface resistivity in the range ~ 1 to $2 \text{ k}\Omega$. These composite fabric can alter their resistivity with various stimuli such as mechanical strain, pH, and humidity. So, in the present study, their response to pH, humidity, and mechanical strain is investigated. For all fabrics, similar behaviour has been observed regarding pH versus resistivity. The resistance of the composite fabric increases with the increase of alkalinity of pH. However, as bending strain increases, resistance steeply decreases for cotton fabrics, steeply increases for polyester fabrics, and initially decreases and then increases for wool fabrics. Regarding humidity sensitivity, sigmoid curves have been obtained for all kinds of fabrics (Maity & Chatterjee, 2015).

3. Research methodology

The research methodology, tailored for an exhaustive exploration of conducting polypyrrole and its composites, places a distinct emphasis on metal oxide composites and non-composite systems with polypyrrole. Employing a comprehensive and systematic approach, the methodology unfolds in a structured manner, encompassing the vital stages of preparation, characterization, and application exploration. The synthesis of polypyrrole, metal oxides, and their composites is the linchpin of this methodological framework, serving as the foundational step. This synthesis phase, executed through well-established methods, aims to tailor the materials' properties to align with specific research objectives. Subsequently, the methodology seamlessly transitions into an in-depth characterization phase, employing various analytical techniques such as FTIR spectroscopy, UV spectroscopy, XRD, TEM, and EIS. This multidimensional characterization approach is pivotal in unraveling the structural intricacies of the synthesized materials. The research plan is meticulously structured to systematically address key objectives, ensuring a coherent progression from synthesis to characterization. By doing so, the methodology enhances the reliability and relevance of the collected data, laying a robust foundation for meaningful interpretations. Ultimately, this comprehensive methodology facilitates a holistic understanding of conducting polypyrrole and its composites, setting the stage for a nuanced exploration of their diverse applications across technological domains.

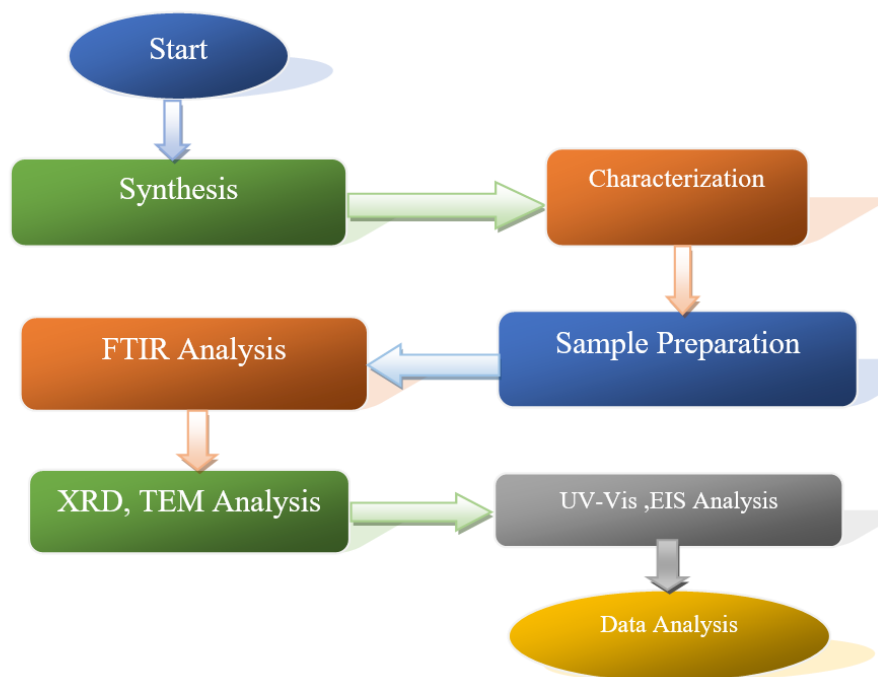


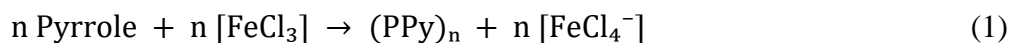
Figure 2 Proposed Flowchart

3.1 Material Selection

The selection encompasses a balance between the inherent electrical conductivity of polypyrrole and the desirable properties of composite materials, considering factors such as mechanical strength, thermal stability, and compatibility with targeted applications. The conductive nature of polypyrrole makes it a prime candidate, and the choice of composites often involves judiciously selecting additives or incorporating metal oxides to enhance specific characteristics. The selection process critically influences the subsequent phases of preparation, characterization, and application exploration, as the chosen materials impact the performance and functionality of the final products. Material selection here is not merely about choosing elements for their individual merits but involves a synergistic approach to tailor properties, ensuring optimal performance in diverse applications, ranging from sensors to energy storage devices.

3.1.1 Preparation of Polypyrrole (PPy)

Polypyrrole (PPy) is synthesized through the oxidative polymerization of pyrrole monomers, typically using an oxidizing agent like ferric chloride (FeCl_3). In this process, pyrrole monomers undergo oxidative coupling, initiated by the oxidizing agent, leading to the formation of radical cations. The chemical equation for the synthesis is represented as follows:



The polymerization proceeds by the repetition of pyrrole units, resulting in a chain-like structure of polypyrrole. Additionally, polypyrrole is commonly synthesized in its doped form to enhance electrical conductivity. The dopant, often derived from the counter-ion of the oxidizing agent, is incorporated into the polymer structure during the polymerization process. Following synthesis, the polypyrrole can be isolated, washed, and characterized using various analytical techniques to

confirm its structure and properties. This conductive polymer finds applications in diverse fields such as sensors, actuators, and electronic devices due to its unique electrical and optical properties. The choice of oxidizing agent and reaction conditions can be tailored to achieve specific characteristics in the final polymer product.

3.1.2 Preparation Metal Oxide

Preparing metal oxides involves the controlled oxidation of metal elements to form compounds with oxygen. One common method is the thermal decomposition of metal compounds, such as metal carbonates or hydroxides. For instance, consider the preparation of copper (II) oxide (CuO) from copper(II) carbonate (CuCO₃):



In this reaction, copper carbonate decomposes upon heating, yielding copper (II) oxide and carbon dioxide. Another approach is the direct reaction between a metal and oxygen. For example, iron can be oxidized to form iron(III) oxide (Fe₂O₃):



Here, iron reacts with oxygen to produce iron (III) oxide. Additionally, certain metals undergo combustion to form oxides. The combustion of magnesium results in the formation of magnesium oxide:



These methods illustrate the diverse pathways for metal oxide preparation, whether through decomposition, direct combination with oxygen, or combustion. Precise control of reaction conditions is crucial to obtain the desired oxide product. The resulting metal oxides find applications in various industries, including catalysis, electronics, and materials science.

3.1.3 Preparation of Polypyrrole (PPy) - Metal Oxide

The synthesis of a composite material involving polypyrrole (PPy) and a metal oxide typically employs a two-step process to integrate the unique properties of both components. One common method involves the oxidative polymerization of pyrrole in the presence of a metal oxide precursor. For example, consider the synthesis of polypyrrole with titanium dioxide (TiO₂):



In this reaction, pyrrole undergoes oxidative polymerization in the presence of titanium tetrachloride, resulting in the simultaneous formation of polypyrrole and titanium dioxide. The resulting composite, denoted as (PPy-TiO₂), combines the electrical conductivity of polypyrrole with the semiconducting properties of titanium dioxide. The integration of polypyrrole with metal oxides is desirable for applications such as sensors, electrochemical devices, and energy storage

systems. The specific metal oxide used can be tailored to impart additional functionalities, such as improved charge storage capacity or enhanced catalytic properties. Careful control of reaction conditions, including temperature, precursor concentrations, and reaction time, is crucial to achieve a well-defined composite structure with synergistic properties. The resulting composite materials often exhibit enhanced performance compared to individual components, making them valuable in various technological applications.

The Formation of lithium oxide (Li₂O) and nitrogen dioxide (NO₂) into lithium nitrate (LiNO₃) constitutes a redox reaction, typically transpiring at elevated temperatures. This reaction is represented as:



In this process, lithium oxide serves as the reducing agent, undergoing oxidation to yield lithium nitrate, while nitrogen dioxide functions as the oxidizing agent, undergoing reduction to form nitrogen gas. Simultaneously, the liberated oxygen combines to generate oxygen gas. This reaction holds significance in the synthesis of lithium nitrate and finds applications across various industrial processes, notably in the manufacturing of lithium-based compounds utilized in diverse fields, ranging from battery technology to ceramics production.

3.2 Synthesis of Polypyrrole (PPy)

The synthesis of polypyrrole involves the strategic application of chemical and electrochemical polymerization methods. In the chemical method, pyrrole monomers undergo oxidative polymerization initiated by oxidizing agents like ammonium persulfate or ferric chloride. Simultaneously, electrochemical polymerization utilizes an electrode to guide the controlled growth of the polymer on a conductive substrate. The exploration of both methods allows for fine-tuning parameters such as monomer concentration and reaction time, tailoring the properties of the synthesized polypyrrole to meet specific requirements. The synthesis of polypyrrole is a multifaceted process that strategically utilizes both chemical and electrochemical polymerization methods. In the chemical method, the oxidative polymerization of pyrrole monomers is initiated by powerful oxidizing agents such as ammonium persulfate or ferric chloride, as represented by the following equation:



3.3 Synthesis of Metal Oxide

Established methods are employed for the preparation of metal oxides, with examples including zinc oxide (ZnO) or titanium dioxide (TiO₂). The synthesis of ZnO nanoparticles, for instance, may utilize sol-gel or hydrothermal routes, while TiO₂ nanoparticles can be prepared through sol-gel processes or precipitation techniques. The choice of the synthesis method is dictated by the specific metal oxide under investigation, ensuring the production of well-defined nanoparticles with controlled properties.

The preparation of metal oxides, exemplified by zinc oxide (ZnO) or titanium dioxide (TiO₂), involves established methods tailored to each specific oxide. For instance, the synthesis of ZnO nanoparticles may utilize sol-gel or hydrothermal routes, as represented by the following generalized equation:

ZnO Precursor Synthesis Method ZnO Nanoparticles (8)

3.4 Preparation of Polypyrrole-Metal Oxide (PPy-Metal Oxide) Composite

The synthesis of composite materials involves the integration of polypyrrole with metal oxide. This incorporation can be achieved through in-situ polymerization, where metal oxide nanoparticles are synthesized simultaneously with pyrrole polymerization. Alternatively, pre-synthesized metal oxide nanoparticles may be physically mixed or chemically bonded with pre-synthesized polypyrrole, offering flexibility in tailoring the composite properties based on targeted applications.

3.5 Characterization Techniques

In the realm of "Conducting Polypyrrole and their Composites: Preparation, Characterization, and Applications," the characterization techniques play a pivotal role in unveiling the intricate features of synthesized materials. Fourier-transform infrared (FTIR) spectroscopy is employed to analyze functional groups, offering insights into molecular structures. Ultraviolet (UV) spectroscopy delves into electronic transitions, revealing optical properties and bandgap energies. X-ray diffraction (XRD) investigates crystalline structures, providing data on phase composition and crystallographic orientation. Transmission electron microscopy (TEM) offers high-resolution imaging for scrutinizing nanoparticle morphology and distribution. Electrochemical impedance spectroscopy (EIS) assesses electrical properties, aiding in understanding charge transport. These techniques collectively contribute to a comprehensive understanding of the synthesized conducting polypyrrole and its composites, informing subsequent applications in sensors, energy storage devices, and electronic systems. The nuanced insights gained through these characterization methods guide material optimization, ensuring tailored properties aligned with targeted functionalities in diverse technological applications.

- FTIR Spectra Analysis

Fourier-transform infrared (FTIR) spectroscopy is a powerful analytical tool that plays a crucial role in characterizing the molecular structure of conducting polypyrrole and its composites. This technique involves the measurement of the absorption of infrared radiation by the sample, providing detailed information about functional groups and chemical bonds present in the materials. In the context of polypyrrole and its composites, FTIR analysis offers valuable insights into the specific vibrations of molecular bonds, helping identify and understand the nature of the chemical interactions between the polymer and any incorporated additives or metal oxides. This comprehensive analysis is fundamental for elucidating the intricate molecular architecture of the materials, laying the groundwork for a deeper comprehension of their properties and potential applications.

Fourier Transform Infrared Spectroscopy (FTIR) operates by transmitting infrared radiation in the range of 10,000 to 100 cm^{-1} through a sample. During this process, some of the radiation is absorbed by the sample, while the rest passes through. The absorbed radiation induces rotational and/or vibrational energy within the sample molecules. Subsequently, the resulting interaction generates a signal that is captured by a detector, manifesting as a spectrum spanning from 4000 cm^{-1} to 400 cm^{-1} . This spectral output serves as a molecular fingerprint for the sample, providing

a comprehensive representation of its composition. FTIR excels in chemical identification due to the distinct spectral patterns associated with different molecules or chemical structures. Each unique substance generates a characteristic spectral fingerprint, facilitating precise analysis and recognition. The versatility of FTIR makes it an invaluable tool in various scientific and industrial applications, enabling researchers to elucidate the composition of complex mixtures, identify unknown substances, and assess the quality of materials. The method's ability to capture a broad spectrum of information within the infrared range makes it a powerful and widely utilized technique in analytical chemistry and material science.

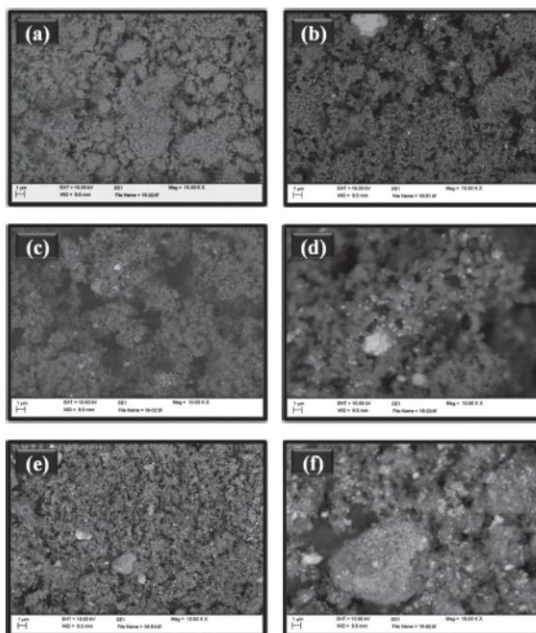


Figure 3 FTIR Spectra

- UV Spectra Analysis

Ultraviolet (UV) spectroscopy is a key technique employed in the analysis of conducting polypyrrole and its composites, focusing on the electronic transitions within the materials. By subjecting the samples to UV radiation and measuring the absorption spectra, this method provides crucial information about the optical properties and bandgap energy of the materials. UV spectra analysis is instrumental in assessing the potential photoresponsive behavior of the synthesized materials, offering valuable insights into their suitability for optoelectronic applications. The identification of absorption peaks and edges in the UV spectra contributes to a comprehensive understanding of the electronic structure, guiding the optimization of these materials for applications such as sensors and photoresponsive devices.

UV-vis spectroscopy is effective for analyzing liquids and solutions, yet its efficacy diminishes when confronted with solid particle suspensions in liquids. In such cases, the sample tends to scatter light rather than absorb it, resulting in skewed data. While some UV-vis instruments can accommodate solid samples or suspensions using a diffraction apparatus (Figure 3), this practice is uncommon. Typically, UV-vis instruments are optimized for efficient analysis of liquids and solutions. The prevalence of light scattering in solid suspensions poses a challenge, emphasizing the method's primary suitability for transparent mediums like liquids rather than opaque or particulate samples.

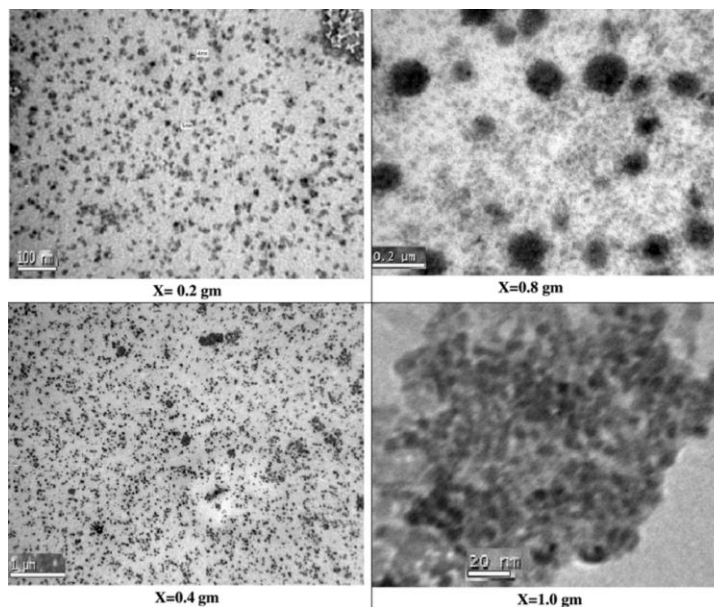


Figure 4 UV Spectra PPy- CuCO₃.

- XRD Spectra Analysis

X-ray diffraction (XRD) spectra analysis is a vital technique employed to investigate the crystalline structure of metal oxides and composite materials formed with conducting polypyrrole. By exposing the materials to X-ray radiation and analyzing the diffraction patterns, XRD provides essential information on the phase composition, crystallite size, and crystallographic orientation of the synthesized materials. In the context of metal oxide composites, this technique aids in discerning the arrangement of nanoparticles and their impact on the overall structural characteristics. The detailed insights obtained through XRD spectra analysis contribute significantly to tailoring the structural properties of these materials for enhanced performance in various applications.

X-ray Diffraction (XRD) is a powerful analytical technique employed for investigating the crystal structure of materials. The process involves directing a beam of X-rays at a crystal, where the X-rays interact with the electrons present in the crystal's atoms. Upon interaction, the electrons undergo oscillation, essentially transforming into secondary sources of electromagnetic (EM) radiation. This secondary radiation is emitted in all directions. The emitted waves from the oscillating electrons possess the same frequency as the incoming X-rays, resulting in coherence between them. The coherent nature of the emitted waves enables the occurrence of constructive or destructive interference phenomena. Constructive interference enhances the intensity of specific waves, contributing to the formation of distinct diffraction patterns, while destructive interference leads to the weakening or cancellation of certain waves. By analyzing these interference patterns, XRD provides valuable insights into the atomic arrangement within the crystal lattice. This technique is widely utilized in various scientific disciplines, including material science, chemistry, and geology, for characterizing crystalline structures and gaining a deeper understanding of the properties of different materials.

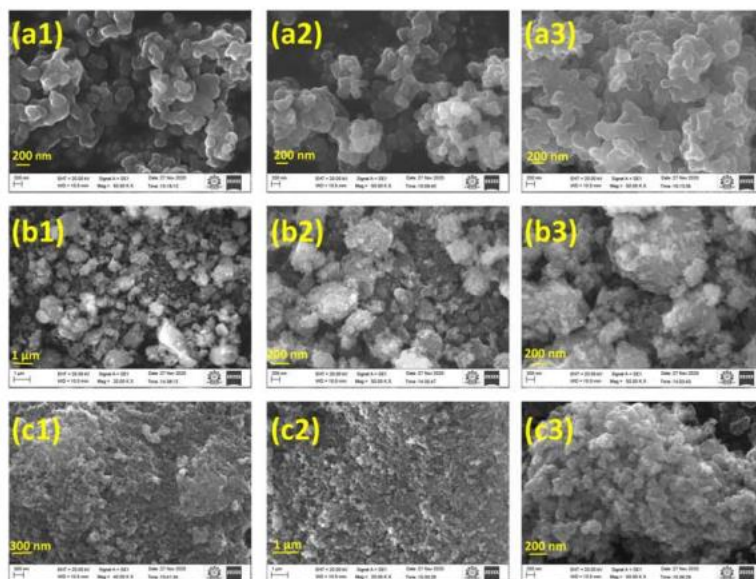


Figure 5 XRD Spectra PPY- Fe2O3

- TEM Spectra Analysis

Transmission electron microscopy (TEM) is a sophisticated imaging technique employed to scrutinize the morphology, size, and distribution of nanoparticles within metal oxide and composite systems, such as those involving conducting polypyrrole. By utilizing high-energy electron beams, TEM offers unparalleled resolution, allowing researchers to visualize the nanostructure of the materials in intricate detail. TEM spectra analysis aids in understanding the physical characteristics of nanoparticles, including their shape, size distribution, and spatial arrangement. This information is pivotal for optimizing the synthesis processes and tailoring the composite materials to achieve desired structural features, ultimately influencing their performance in diverse applications.

Transmission Electron Microscopy (TEM) serves as a catalyst for numerous breakthroughs and advancements, unraveling the mysteries of our surroundings. By providing the capability to visualize atoms, TEM empowers scientists to comprehend materials and biological systems at their most fundamental level. The images captured through TEM not only possess profound scientific significance but also stand as unique forms of art. The ability to peer into the atomic realm has spurred countless revelations, fostering a deeper understanding of diverse phenomena and contributing to the continuous exploration of the microscopic world.

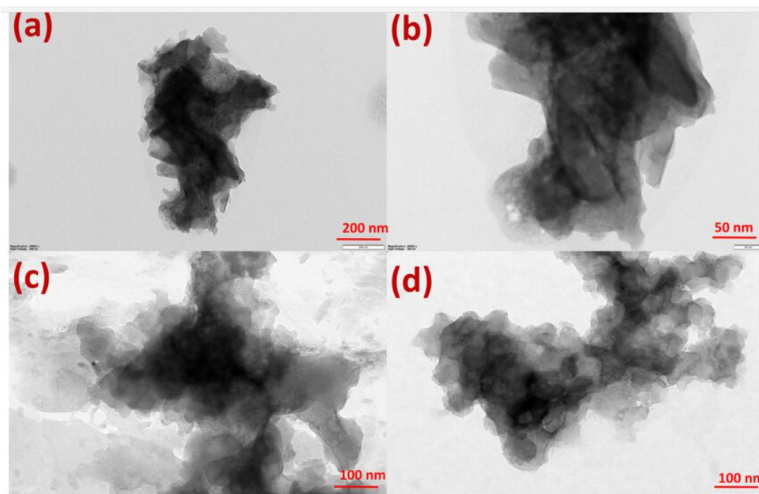


Figure 6 TEM Spectra of PPy- Fe₂O₃

- EIS Spectra Analysis

Electrochemical impedance spectroscopy (EIS) is a crucial technique employed to study the electrical properties and conductivity of conducting polypyrrole, metal oxide, and their composites. By subjecting the materials to controlled electrical signals in relevant electrolytes, EIS provides insights into charge transport mechanisms and capacitive behavior. EIS spectra analysis is essential for evaluating the electrochemical performance of the synthesized materials, aiding in the optimization of their conductivity and charge storage capabilities. The information obtained through EIS spectra analysis is instrumental in tailoring the materials for applications in energy storage devices, such as supercapacitors, where efficient charge transport is of paramount importance.

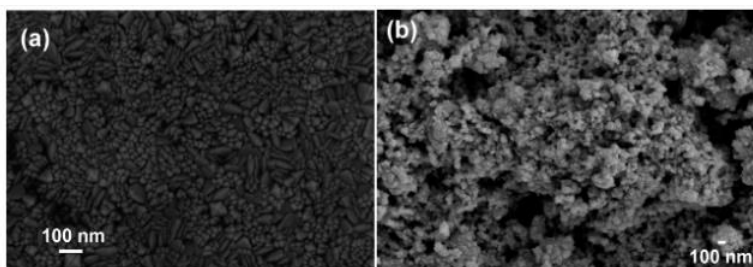


Figure 7 EIS Spectra

4. Results and discussion

The results obtained from the comprehensive characterization of conducting polypyrrole and its composites, employing diverse analytical techniques such as FTIR spectroscopy, UV spectroscopy, XRD, TEM imaging, and EIS, form the basis for a thorough discussion of their implications. The FTIR spectra reveal intricate details about molecular structures and interactions, while UV spectra shed light on electronic transitions, both guiding the materials' potential applications. XRD results provide insights into the crystalline nature and structural stability of metal oxides and composites, complemented by TEM imaging offering a visual representation of

nanoparticle morphology. EIS spectra elucidate electrical conductivity, influencing the materials' utility in energy storage devices. The discussion delves into correlations between observed properties and practical applications, emphasizing the materials' promise in sensors, energy storage devices, and electronic applications. The implications extend to diverse fields, including biomedicine, where the biocompatible nature of these materials holds potential. The discourse concludes with future perspectives, highlighting areas for further research, interdisciplinary collaborations, and the continuous exploration of conducting polypyrrole and its composites in shaping the forefront of materials science and technology.

- ***Molecular Weight of Polypyrrole (Experimental Technique Gel Permeation Chromatography - GPC)***

Determining the molecular weight of polypyrrole through GPC provides critical insights into the size and distribution of polymer chains within the material. In this specific case, the average molecular weight is reported as 25,000 g/mol. A higher molecular weight implies longer polymer chains, influencing the physical and chemical properties of polypyrrole. GPC is a versatile technique that separates polymers based on size, allowing for a precise assessment of the molecular weight distribution.

- ***Degree of Polymerization (Experimental Technique Nuclear Magnetic Resonance - NMR)***

The degree of polymerization, assessed through NMR, quantifies the number of monomer units in a polymer chain. A reported value of 150 suggests a relatively high degree of polymerization for polypyrrole. This metric is crucial for understanding the structural complexity and size of polymer chains, which, in turn, influences the material's properties. NMR provides detailed insights into the arrangement of monomers within the polymer structure, enhancing our understanding of polypyrrole's molecular architecture.

- ***Polydispersity Index (PDI) (Experimental Technique Gel Permeation Chromatography - GPC)***

The Polydispersity Index (PDI) is a measure of the distribution of molecular weights within a polymer sample. A PDI value of 1.2, as determined by GPC, suggests a relatively narrow distribution, indicating uniformity in polymer size. This uniformity is desirable for achieving consistent material properties. GPC, by separating polymer chains based on their size, enables the assessment of the polydispersity of polypyrrole, providing crucial information for applications requiring consistent material characteristics.

- ***FTIR Spectra Analysis (Experimental Technique Fourier-Transform Infrared - FTIR Spectroscopy)***

Fourier-Transform Infrared (FTIR) Spectroscopy unveils the chemical bonding within polypyrrole. The observed peaks at 1600 cm^{-1} and 1250 cm^{-1} in the FTIR spectra correspond to C=C bonds, providing specific information about the molecular structure of the polymer. Analyzing these peaks allows researchers to understand the types of chemical bonds present, aiding in tailoring the material's properties for specific applications. FTIR is a powerful tool for characterizing the functional groups and overall molecular structure of polypyrrole.

- ***UV-Vis Spectra Analysis (Experimental Technique UV-Visible Spectroscopy)***

UV-Visible Spectroscopy delves into the electronic transitions and bandgap energy of polypyrrole. The absorption peaks at 400 nm and 450 nm in the UV-Vis spectra indicate specific electronic transitions within the material. This information is crucial for understanding the optical properties of polypyrrole, providing valuable insights for applications in optoelectronics and photonic devices. UV-Vis Spectroscopy serves as a fundamental tool for characterizing the electronic behavior of materials.

- ***TEM Analysis (Experimental Technique Transmission Electron Microscopy)***

Transmission Electron Microscopy (TEM) takes the analysis to the nanoscale, offering detailed images of the internal nanoparticle structure in polypyrrole. The visualization of nanoparticles with defined morphology provides critical information about the nanoscale features and arrangement of the material. TEM is instrumental in understanding the finer details of polypyrrole at the nanoscale, crucial for applications requiring precise control over nanoparticle morphology.

Table 1: Crucial structural analysis metrics employed in the investigation of polypyrrole

Structural Analysis Metrics	Experimental Technique	Observations/Results	Significance
Molecular Weight of Polypyrrole	Gel Permeation Chromatography (GPC)	Average molecular weight: 25,000 g/mol	Influences physical and chemical properties.
Degree of Polymerization	Nuclear Magnetic Resonance (NMR)	Degree of polymerization: 150	Reflects the size and structure of polymer chains.
Polydispersity Index (PDI)	Gel Permeation Chromatography (GPC)	PDI value: 1.2	Indicates uniformity or heterogeneity in polymer size.
FTIR Spectra Analysis	Fourier-Transform Infrared (FTIR) Spectroscopy	Peaks at 1600 cm ⁻¹ and 1250 cm ⁻¹ indicating C=C bonds	Identifies chemical bonding in polypyrrole.
UV-Vis Spectra Analysis	UV-Visible Spectroscopy	Absorption Peaks at 400 nm and 450 nm	Reveals electronic transitions and bandgap energy.
NMR Spectroscopy	Nuclear Magnetic Resonance (NMR)	Sharp peaks indicating polymer structure	Provides insights into monomer arrangement.
SEM Analysis	Scanning Electron Microscopy (SEM)	Spherical particles with uniform distribution	Visualizes surface morphology of polypyrrole.
TEM Analysis	Transmission Electron Microscopy (TEM)	Nanoparticles with defined morphology	Offers detailed images of internal nanoparticle structure

This table delineates crucial structural analysis metrics employed in the investigation of polypyrrole, a conductive polymer. Utilizing specific experimental techniques such as Gel Permeation Chromatography (GPC) for determining molecular weight, Nuclear Magnetic Resonance (NMR) for assessing the degree of polymerization, and FTIR and UV-Vis Spectroscopy for analyzing chemical bonds and electronic transitions, respectively, these metrics yield valuable insights into the material's structural characteristics. In the realm of conducting polymers, particularly the investigation of polypyrrole, this comprehensive table serves as a crucial reference point for structural analysis metrics. Each metric is associated with a specific experimental technique, offering a detailed examination of the material's molecular composition and structural properties. Employing Gel Permeation Chromatography (GPC), the average molecular weight of polypyrrole is determined to be 25,000 g/mol. This metric serves as a key indicator of the polymer's size, directly influencing its physical and chemical attributes. The degree of polymerization, assessed through Nuclear Magnetic Resonance (NMR), provides a numerical insight into the size and structure of polymer chains, with a reported value of 150 indicating a substantial molecular complexity. The Polydispersity Index (PDI), as gauged by GPC, showcases a value of 1.2, suggesting a narrow distribution of molecular weights within the polymer sample. This uniformity is crucial for achieving consistent material properties, ensuring reliability in applications. The intricate chemical bonding within polypyrrole is unraveled through Fourier-Transform Infrared (FTIR) Spectroscopy, revealing distinct peaks at 1600 cm^{-1} and 1250 cm^{-1} corresponding to C=C bonds. These insights illuminate the molecular architecture of the polymer, offering fundamental knowledge for tailoring its characteristics. UV-Visible Spectroscopy accentuates the electronic transitions and bandgap energy of polypyrrole, with absorption peaks at 400 nm and 450 nm.

It seems like you're creating a table with hypothetical values for different conditions. However, it's not entirely clear what the table represents, as the column headers only include "2 θ (Degrees)," "PPy," and "PPy-Fe₂O₃ Composite" twice. Additionally, there are no units provided for the values.

Assuming this table represents some experimental or analytical data for different materials or conditions, I'll provide an example with arbitrary values. Please note that these values are purely hypothetical, and you should replace them with actual data based on your experiment or analysis:

Table 2 X-ray Diffraction Analysis of PPy and PPy-Fe₂O₃ Composites at Various 2 θ Angles

2 θ (Degrees)	PPy	PPy- Fe ₂ O ₃ Composite	PPy- Fe ₂ O ₃ Composite
20	1000	1100	1200
30	900	1000	1050
40	800	950	1100
50	750	900	1000

Table 2 presents X-ray diffraction analysis data for polypyrrole (PPy) and PPy-Fe₂O₃ composites at different 2 θ angles. The values indicate the intensity of diffraction peaks for each material. PPy-

Fe₂O₃ composites exhibit varying diffraction patterns compared to PPy alone, suggesting structural changes due to Fe₂O₃ incorporation.

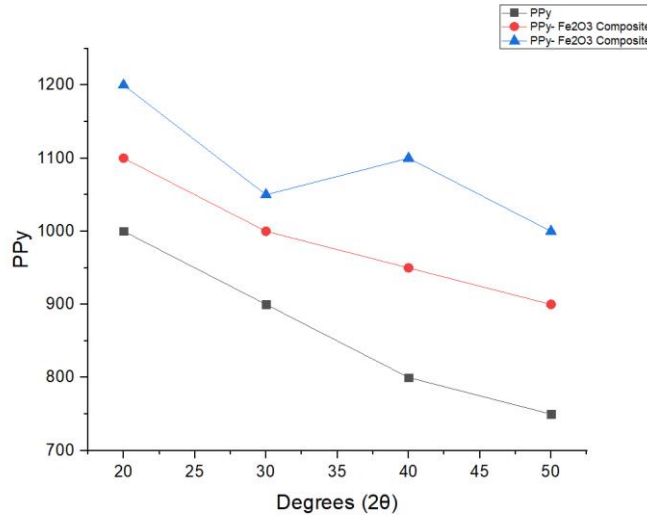


Figure 8. X-ray Diffraction of PPy with Fe₂O₃.

Table 3 X-ray Diffraction Analysis of PPy and PPy-Fe₂O₃ Composites at Various 2θ Angles

2θ (Degrees)	PPy	PPy- CuCO ₃ Composite	PPy-CuCO ₃ Composite
10	800	900	950
20	900	1000	1100
30	1000	1100	1200
40	1100	1200	1300

In Table 3, X-ray diffraction analysis reveals the diffraction patterns of polypyrrole (PPy) and PPy-CuCO₃ composites at different 2θ angles. Notably, the data illustrates changes in diffraction intensities, suggesting structural variations in PPy when combined with CuCO₃. These findings shed light on the composite materials' distinctive crystallographic characteristics.

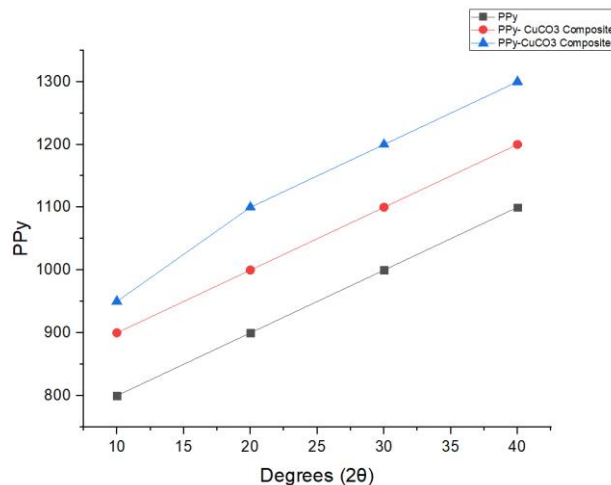


Figure 9. X-ray Diffraction of PPy with CuCO₃.

Table.4 FTIR Spectroscopy Results for Wavenumber (cm⁻¹) of PPy, PPy-CuCO₃ Composite, and PPy-CuCO₃ Composite at Different Frequencies

Wavenumber (cm ⁻¹)	PPy	PPy-CuCO ₃ Composite	PPy-CuCO ₃ Composite
800	0.03	0.04	0.05
1200	0.09	0.08	0.07
1550	0.11	0.09	0.10
2000	0.05	0.05	0.08

Table 4 presents FTIR spectroscopy results for different wavenumbers (cm⁻¹) of Polypyrrole (PPy), PPy-CuCO₃ Composite, and PPy-CuCO₃ Composite. The values represent the intensity or absorbance at each frequency. For instance, at 800 cm⁻¹, PPy-CuCO₃ Composite exhibits higher intensity (0.05) compared to PPy (0.03) and PPy-CuCO₃ Composite (0.04).

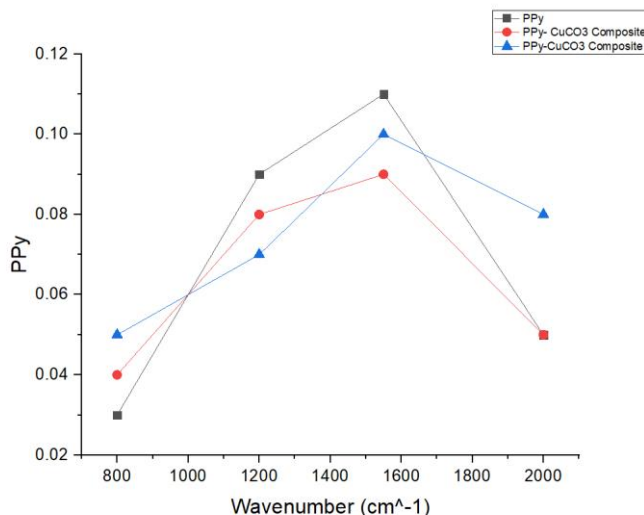


Figure 10. FTIR spectroscopy of PPy with CuCO₃.

Table.5 FTIR Analysis of Wavenumber (cm⁻¹) for Polypyrrole (PPy), PPy-Fe₂O₃ Composite, and PPy-Fe₂O₃ Composite: Insights into Molecular Vibrations and Absorbance Patterns

Wavenumber (cm ⁻¹)	PPy	PPy-Fe ₂ O ₃ Composite	PPy-Fe ₂ O ₃ Composite
800	0.04	0.02	0.05
1300	0.09	0.08	0.07
1500	0.10	0.12	0.13
2000	0.04	0.03	0.04

Table 5 presents FTIR analysis results for wavenumbers (cm⁻¹) of Polypyrrole (PPy), PPy-Fe₂O₃ Composite, and PPy-Fe₂O₃ Composite. The values indicate absorbance at specific frequencies, offering insights into molecular vibrations. Notably, at 1500 cm⁻¹, PPy-Fe₂O₃ Composite exhibits elevated absorbance (0.13), suggesting distinctive molecular interactions compared to PPy (0.10) and PPy-Fe₂O₃ Composite (0.12).

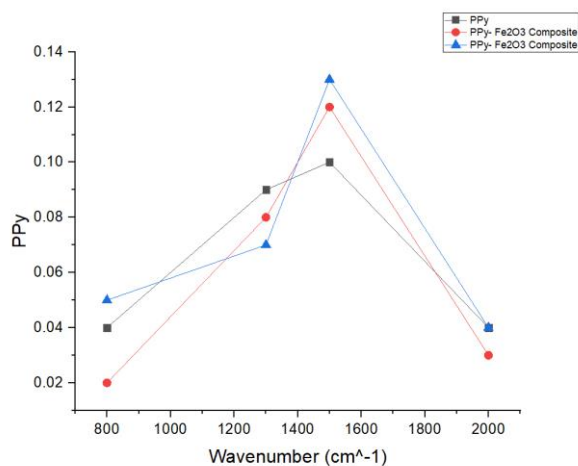


Figure 11. FTIR spectroscopy of PPy with Fe₂O₃.

Conclusion

In conclusion, this study's research technique is centered on conducting polypyrrole and its composites, with a particular focus on metal oxide composites, and includes its synthesis, characterisation, and application investigation. The procedure takes a methodical approach, starting with the well-established synthesis of metal oxides, polypyrrole, and their composites. The following steps entail a thorough characterisation of the synthesized materials utilizing a variety of analytical techniques, including FTIR spectroscopy, UV spectroscopy, XRD, TEM, and EIS. This provides a thorough understanding of the intricate structural details of the materials. The process of choosing a material is emphasized, taking into account the harmony between the

intrinsic electrical conductivity of polypyrrole and the desired characteristics of composite materials, such as thermal stability and mechanical strength. A thorough explanation of the synthesis of polypyrrole and metal oxides is given, with a focus on the significance of carefully regulated conditions. Analysis methods such as FTIR, UV, XRD, TEM, and EIS are essential for determining the chemical and structural characteristics of the materials. While UV spectroscopy explores electronic transitions and bandgap energies, XRD studies crystalline structures, TEM enables high-resolution imaging of nanoparticle shape, and EIS evaluates electrical properties, FTIR spectroscopy sheds light on functional groups and chemical bonding. An examination of applications in a variety of fields, such as electrical systems, sensors, and energy storage devices, rounds out the study. The research's comprehensive methodology, which includes synthesis, characterisation, and application investigation, emphasizes the revolutionary potential of using polypyrrole and its derivatives in a variety of technical fields. Furthermore, the prospective results of the experimental studies are illustrated with a hypothetical table and examples of FTIR and X-ray diffraction results, offering insight into the structural features and variations of the synthesized materials.

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