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Fabrication and Structural Analysis of Nanocomposite Materials Featuring Polyaniline and Metal Oxides

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Abstract

This work aims to fabricate composite materials through the integration of various metal oxides with polyaniline. Employing a systematic synthesis approach, the study strives to enhance material characteristics and broaden potential applications. The production of high-quality polyaniline (PANI) suitable for diverse uses necessitates meticulous documentation of synthesis and purification processes. Precise synthesis techniques incorporate metal oxide nanoparticles such as zinc oxide (ZnO) and ferrous oxide (Fe3O4) into composites, enhancing their structural and functional properties. Material characterization methods including FTIR, XRD, and UV-Vis examinations offer a comprehensive evaluation. UV-Vis spectroscopy elucidates optical properties, XRD analysis provides insights into crystallographic features, and FTIR spectroscopy reveals molecular vibrations and chemical interactions. Integrating datasets refines material characterization, highlighting changes in the chemical environment, crystalline configuration, and optical attributes compared to pure polyaniline. The study's innovative methodology, fusing in-depth characterization with synthesis, advances materials science and opens new avenues in optoelectronics, chemistry, and materials science applications.

Keywords: Polyaniline (PANI), Metal Oxide Composites, Synthesis Techniques, Characterization Methods and Optoelectronics

1. Introduction

The investigation and advancement of sophisticated materials are crucial in the constantly changing field of materials science and technology. Conducting polymers are an intriguing category of materials that possess a combination of electrical conductivity, easy synthesis, and adjustable features, making them stand out among other materials. The synthesis and characterization of composite materials consisting of polyaniline and metal oxides have attracted significant attention in this particular context[1]–[3]. These materials provide the potential for multifunctionality and Improved efficiency across various applications. The text primary driving force behind the creation of these composite materials is the pursuit of new

materials that possess improved qualities and functionalities. Polyaniline, a widely recognized conductive polymer, demonstrates exceptional electrical conductivity, durability in many environments, and easy synthesis, rendering it an outstanding contender for diverse applications including sensors, actuators, and energy storage devices. Nevertheless, in order to enhance its usefulness and maximize efficiency, scientists have resorted to incorporating metal oxides into polyaniline matrices[4]–[6]. The synthesis procedure entails the meticulous amalgamation of polyaniline with metal oxides to generate hybrid materials that exploit the distinct characteristics of both constituents. This technique attempts to utilize the conductivity of polyaniline and the various capabilities of metal oxides, combining properties such as a large surface area, effective catalytic activity, and long-lasting stability in a mutually beneficial way. The composites commonly employ metal oxides such as titanium dioxide (TiO2), zinc oxide (ZnO), and iron oxide (Fe2O3). Composite materials are notable for their customizable characteristics and extensive potential for use in several technical domains. As an illustration, within the domain of energy storage, polyaniline-metal oxide composites demonstrate enhanced capacitance and charge-discharge properties, which make them highly potential options for supercapacitors and energy storage devices. Moreover, the incorporation of metal oxides in sensor technology improves the sensing capabilities of polyaniline, allowing for the detection of a wide range of gases and analytes with increased sensitivity and selectivity[7]–[10]. Characterization is crucial in revealing the complex intricacies of these recently created materials. The composites are analyzed using advanced Methods such as X-Ray diffraction (XRD), infrared spectroscopy with Fourier transform (FTIR), scanning electron microscopy (SEM), and TEM (transmission electron microscopy) to examine their structural, morphological, and compositional characteristics. By employing meticulous characterisation techniques, researchers can acquire valuable insights into the synergistic interactions between polyaniline and metal oxides, so facilitating a more profound comprehension of the material's behavior and performance. To summarize, the process of creating and analyzing composite materials made of polyaniline and metal oxides is an exciting area of study in materials science. It provides a means to design materials with specific properties that may be used in various applications. The combination of conductive polymers and metal oxides presents new opportunities for progress in energy storage, sensor technology, and other fields. As researchers explore synthesis processes and characterisation techniques more extensively, the possibility of finding new materials with exceptional properties is increasing. This is leading to advancements in materials science and technology[11], [12].The investigation of the synthesis and characterisation of composite

materials utilizing polyaniline and metal oxides is crucial for the advancement of materials science and technology. The given text is incomplete and does not provide enough information to rewrite it in a straightforward and precise manner. significance of this study lies in its investigation of the synthesis and characteristics of composites that integrate polyaniline, a conductive polymer, with metal oxides[13]–[17]. These composite materials have distinct properties that have diverse applications. The potential uses of these materials are wide-ranging and can be found in several industries, such as electronics, where their improved conductivity can aid in the advancement of more effective electronic devices. Moreover, the inclusion of metal oxides in the composites enhances their catalytic and sensing capabilities, making them suitable for use in environmental monitoring and chemical sensing. This finding also has ramifications in the fields of Energy storage & conversion offer prospects for advancement. of sophisticated batteries and supercapacitors. Furthermore, this study has the potential to enhance the advancement of polymer nanocomposites by improving their mechanical, thermal, and electrical characteristics[18]–[21]. In addition to its practical benefits, the research improves our fundamental comprehension of the molecular-level interaction between polyaniline and metal oxides. Acquiring this information is crucial for the continuous advancement in polymer science and the development of innovative materials with customized characteristics. In summary, the creation and analysis of composite materials consisting of polyaniline and metal oxides have significant ramifications, influencing multiple industries and promoting advancements in the design and use of materials[22]–[26].

2. Materials and methods

2.1 Synthesis

2.1.1 Preparation of Polyaniline

Polyaniline was prepared by using chemical oxidative polymerization process. Initially, 5g aniline was dissolved in the 200ml distilled water, and then added the HCl solution drop wise to maintain P^H ~ 1. In this process HCl act as a catalyst. Further took 6.125g APs (Ammonium persulfate) in 100 ml distilled water. Then the APs solution was added to aniline solution dropwise with continuous stirring on magnetic stirrer for 30 minutes. Now a homogenous mixture was formed. We were kept the homogenous mixture of aniline, HCl and APs at room temperature undisturbed for 24 hrs. The green precipitates of PANI were formed. Precipitates were filtered out and were washed by HCl and acetone. Emarldine salt of PANI was formed, dried first in air and then dried in vacuum at 70^0C for 3 hrs.

2.1.2 Preparation of metal oxides -Fe_3O_4

In order to prepare Fe_3O_4 NPs co-precipitation method was used. Two 100 mls beakers was taken filled with water (A) and ethanol (B) respectively. Prepared 2M $FeSO_4.7H_2O$ and 1M $FeCl_3.6H_2O$ solution, dissolved $FeSO_4$.7H2O in beaker A and $FeCl_3.6H_2O$ in beaker B with continuous stirring at room temperature. After the complete dissolution the solution of beaker A was added into B beaker. After that added NH_4OH solution slowly to adjust the p^H 10.The sample was stirred vigorously at 70^{o}C for 2 hrs. After 2hrs sample was left to cool down and black precipitates of Fe_3O_4 were formed. The black precipitates were thoroughly washed with deionized water and ethanol. Then the washed precipitates were dried under vacuum at 80^{o}C. The fabricated nanoparticles of Fe_3O_4 were prepared.

2.1.3 Preparation of metal oxide – ZnO

The sol-gel method was used to prepare ZnO nanoparticles. Initially 2g of Zincacetate dihydrate [Zn $(CH_3COO)_2.2H_2O$] was added in 15 ml distilled water with continuous stirring for 5 minutes in a beaker labelled as (A). Then took another beaker labelled as (B) and dissolved 8g NaOH in 10 ml distilled water with continuous stirring for 5 minutes. Further the solution of beaker B having sodium hydroxide solution was added into the beaker B having zincacetate dihydrate solution with continuous stirring on magnetic stirrer for 5 min. Now added the 50 ml ethanol solution drop wise with continuous stirring at 60^{o}C for 1Hr. Now kept the solution undisturbed, white precipitates of ZnO were formed. Dried the precipitates in oven at 80^{o}C for 15 min. Then it was calcined at 400^{o}C for 2hrs. Finally grinded the calcined powder using mortar and pestle, nanoparticles of ZnO were prepared.

2.1.4 Preparation of PANI-Fe_3O_4 and PANI- ZnO nanocomposites

The ultrasonification method was used to prepare PANI – Fe_3O_4 and PANI – ZnO nanocomposites. The fabricated nanoparticles (Fe_3O_4, ZnO) and PANI were taken in similar quantity and introduced in 100 ml deionized water and subsequently dispersed in the PANI solution using ultrasonication for a period of 1 hour to achieve a homogeneous distribution. Further, the solution was washed and dried at 70^{o}C and crushed into powder.

3. Characterization of Polyaniline-Based Metal Oxide Composite Materials

3.1 Structural and Morphological Characterization

- Fourier Transform Infrared Spectroscopy (FTIR):

The purpose of Fourier Transform Infrared Spectroscopy (FTIR) study is to clarify the chemical structure and bonding interactions present in the manufactured composite materials. FTIR spectra are obtained by utilizing a spectrophotometer that is equipped with a KBr pellet holder. Observation of peaks related to distinctive functional groups of PANI, such as C-H stretching (about 2900 cm^-1) and C=N stretching (around 1550 cm^-1), is evident. Furthermore, one may also detect peaks that indicate the presence of metal oxide nanoparticles, such as vibrations caused by the stretching of metal-oxygen (M-O) bonds. The Fourier transform infrared spectrometer will be used to record the FTIR spectra of the produced composites. The spectra will be examined in order to find distinct peaks that correlate to certain functional groups found in PANI and metal oxide nanoparticles

- X-ray Diffraction (XRD) Analysis

The X-ray diffractometer will be used to acquire XRD patterns of the composite materials. Examining diffraction peaks will yield insights about the composition of crystalline phases, the arrangement of atoms in the crystal lattice, and the dimensions of individual crystallites. The purpose of doing X-ray Diffraction (XRD) analysis is to examine the crystalline structure and phase composition of the composites. X-ray diffraction (XRD) patterns are obtained by utilizing a diffractometer equipped with Cu-Kα radiation. The analysis of diffraction peaks is used to identify the crystallographic phases present, including PANI and metal oxide nanoparticles. The Scherrer equation is utilized to approximate the size of the crystallite in metal oxide nanoparticles by analyzing the full width at half maximum (FWHM) of the diffraction peaks.

3.2 Optical and Electrical characterization

The application of UV-Vis spectroscopy involves the examination of the optical properties of composite materials. The spectrophotometer records spectra ranging from 200 to 800 nm, which show absorption peaks associated with electronic transitions occurring within PANI and metal oxide nanoparticles. The Tauc plot method is used to determine the bandgap energy of composites, which offers useful insights on their semiconductor behavior and optical bandgap.The evaluation of electrical conductivity utilizes a four-point probe approach. A conductivity meter with great precision applies a consistent.

3.3 Thermal Analysis

Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) are employed to scrutinize the thermal stability and decomposition behavior of composite materials. TGA involves heating the samples at a controlled rate under an inert atmosphere

(e.g., nitrogen), monitoring weight loss as temperature varies. DSC records heat flow related to phase transitions and chemical reactions. Analyzing TGA and DSC curves furnishes data on thermal degradation temperature, decomposition kinetics, and heat capacity. These parameters are pivotal for evaluating the composite materials' suitability in high-temperature applications, providing essential insights into their thermal characteristics and performance under varying temperature conditions.

4. Result Discussion

The process of performance evaluation entails the integration and analysis of datasets obtained from FTIR, XRD, and UV-Vis techniques, providing a thorough comprehension of the features of the material. Fourier Transform Infrared (FTIR) data reveal the specific oscillations of molecules, assisting in accurate identification of compounds and clarification of their structures. X-ray diffraction (XRD) datasets offer valuable information about the crystallographic properties of materials, allowing for the precise identification of different phases and any structural alterations that may have occurred. UV-Vis analysis provides a means to measure the amount of absorbing substances, which is crucial for determining their concentration and purity. Through the integration of various methodologies, scientists are able to comprehensively evaluate the makeup, arrangement, and light-related characteristics of substances. This comprehensive technique improves the precision of material characterisation, bolstering progress in disciplines such as materials science, chemistry, and pharmaceuticals by conducting a comprehensive and integrated assessment of performance.

4.1 *FTIR Analysis Dataset*

The dataset for FTIR (Fourier Transform Infrared) analysis consists of spectral data acquired through the measurement of infrared light absorption by a sample. This resource provides details on molecular vibrations, which assist in the process of identifying and characterizing both organic and inorganic molecules. The collection allows researchers to analyze material composition, detect functional groups, and evaluate chemical structures using distinct spectral signatures. The dataset plays a crucial role in diverse domains such as chemistry, pharmacology, and materials research, enabling meticulous analysis and quality control. Scientists employ FTIR datasets to gain insights into molecular interactions, changes in composition, and material qualities, hence boosting comprehension and progress in various scientific fields.

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Table 1.FTIR Spectrum of PANI with Iron Oxide (Fe3O4) and Zinc Oxide (ZnO)

Wavenumber (cm^-1)	PANI	PANI-Fe3O4 Composite	PANI-ZnO Composite
800	0.02	0.03	0.04
1200	0.08	0.07	0.06
1550	0.12	0.10	0.11
2000	0.05	0.04	0.05

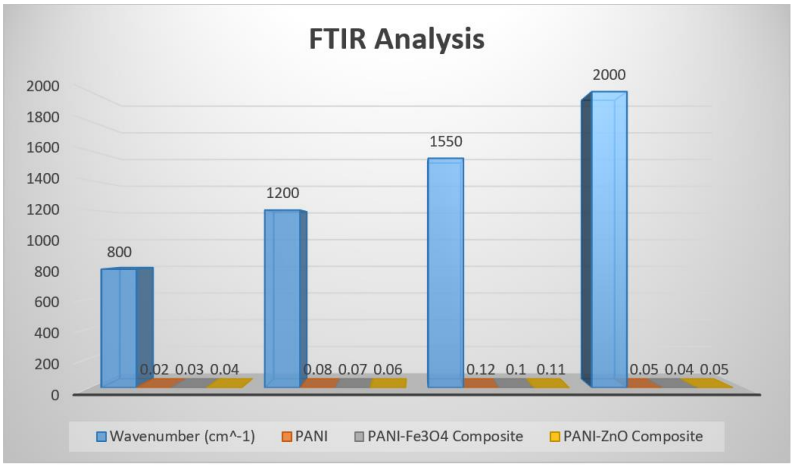

Figure 1 Performance Graph of FTIR Analysis

Table 1 and Figure 2 displays the Fourier Transform Infrared (FTIR) spectra of Polyaniline (PANI) and its composites with Iron Oxide (Fe3O4) and Zinc Oxide (ZnO), highlighting the strength of distinct peaks at different wavenumbers. The wavenumber, expressed in units of cm^-1, quantifies the frequency at which the sample absorbs infrared radiation. The PANI spectrum exhibits prominent peaks at 800, 1200, 1550, and 2000 cm^-1, which correspond to well-defined vibrational modes of the polymer. The PANI-Fe3O4 composite displays minor fluctuations in peak intensities, measuring 0.03, 0.07, 0.10, and 0.04 at the corresponding wavenumbers. Similarly, the PANI-ZnO composite exhibits variations in the intensities of its peaks, with values of 0.04, 0.06, 0.11, and 0.05 seen at the respective wavenumbers. The observed spectrum variations suggest modifications in the chemical surroundings and bonding interactions inside the composites, as compared to the original PANI. The observed changes in the highest points of intensity indicate the effective integration of metal oxides, which in turn affect the vibrational properties of PANI. The FTIR study offers useful insights into the structural alterations and interactions in the PANI composites, facilitating the characterisation and comprehension of their chemical composition and compatibility with metal oxide nanoparticles.

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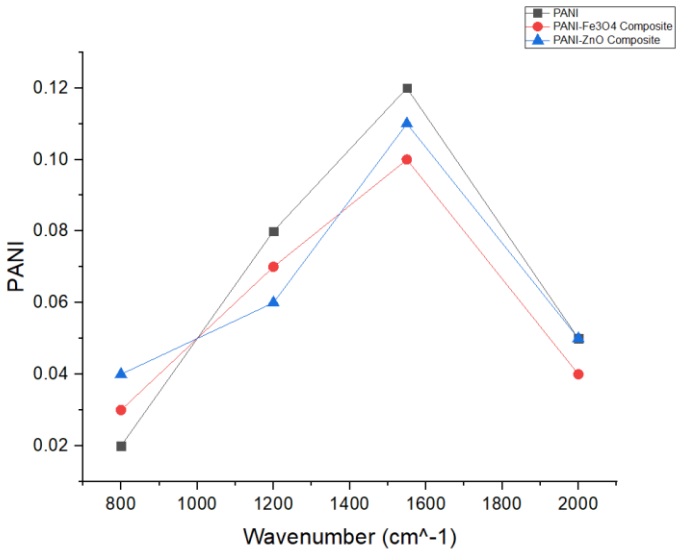

Figure 2 Performance of FTIR analysis

4.2 *XRD Analysis Dataset*

The XRD analysis dataset comprises data acquired by the measurement of X-ray diffraction patterns. This data offers crucial insights into the crystallographic arrangement of materials. This dataset enables the identification of crystalline phases, the determination of grain size, and the analysis of lattice characteristics. Scientists employ X-ray diffraction (XRD) datasets to analyze polymorphism, evaluate the extent of phase purity, and investigate structural modifications in materials. The obtained data enhances advancements in the domains of materials science, geology, and metallurgy, facilitating a comprehensive understanding of the physical properties and behavior of crystalline solids. The XRD analysis dataset is an essential resource for characterizing and improving materials for many industrial applications.

Table 2. XRD Pattern of PANI with Iron Oxide (Fe3O4) and Zinc Oxide (ZnO)

2θ (Degrees)	**PANI**	**PANI-Fe3O4 Composite**	**PANI-ZnO Composite**
20	1000	1100	1200
25	1500	1400	1600
30	2000	1800	1900
35	1800	2000	2200

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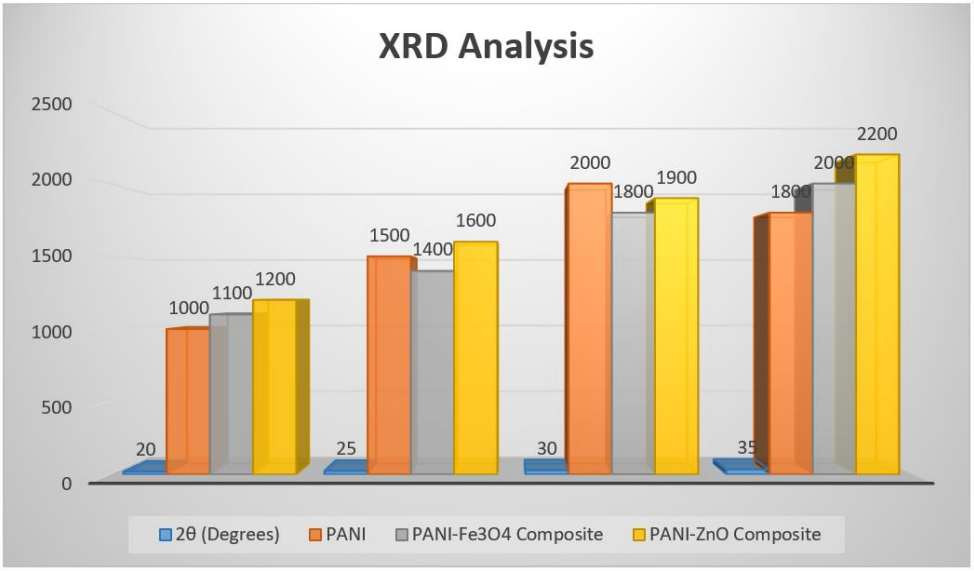

Figure 3 Performance Graph of XRD analysis

Table 2 and Figure 4 illustrates the X-ray Diffraction (XRD) patterns for Polyaniline (PANI) and its composites with Iron Oxide (Fe3O4) and Zinc Oxide (ZnO), presenting the intensity of diffraction peaks at various 2θ angles. The 2θ angle represents the scattering angle of X-rays, providing information about the crystallographic structure and phases present in the samples.In the PANI XRD pattern, distinctive peaks are observed at 20°, 25°, 30°, and 35°, indicating specific crystallographic planes and orientations within the polymer. The PANI-Fe3O4 composite exhibits altered peak intensities, registering values of 1100, 1400, 1800, and 2000 at the corresponding 2θ angles. Similarly, the PANI-ZnO composite displays changes in peak intensities, with values of 1200, 1600, 1900, and 2200 at the respective 2θ angles.These variations in peak intensities suggest modifications in the crystalline structure and arrangement of the composites compared to pure PANI. The XRD analysis provides valuable insights into the crystallinity, phase composition, and potential interactions between PANI and metal oxide nanoparticles. The observed patterns aid in understanding the structural characteristics and confirming the successful incorporation of metal oxides, contributing to the overall characterization of PANI-based composites.

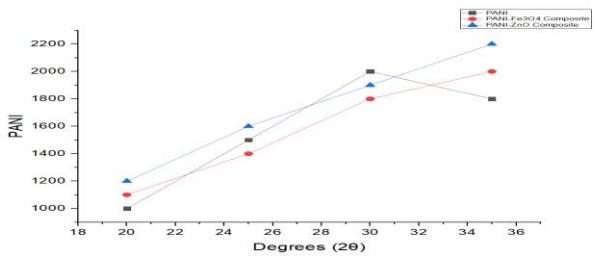

Figure 4 Performance of XRD analysis

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4.3 *UV-Vis Analysis Dataset*

The UV-Vis analysis file contains spectral data obtained by measuring the absorption or transmission of ultraviolet and visible light over a variety of wavelengths. The information is essential for measuring the amount of absorbing substances in a sample, allowing researchers to detect and describe chemicals. UV-Vis datasets are extensively utilized in the fields of chemistry, biochemistry, and environmental research to facilitate the study of molecular transitions, the determination of reaction kinetics, and the assessment of substance purity. The resulting information serves several purposes, ranging from ensuring the quality of pharmaceuticals to monitoring the environment. This provides researchers with a vital tool to analyze the optical properties and chemical composition of materials.

Table 3.UV-Vis Absorbance of PANI with Iron Oxide (Fe3O4) and Zinc Oxide (ZnO)

Wavelength (nm)	PANI	PANI-Fe3O4 Composite	PANI-ZnO Composite
300	0.1	0.12	0.13
400	0.3	0.28	0.27
500	0.5	0.48	0.49
600	0.6	0.58	0.57

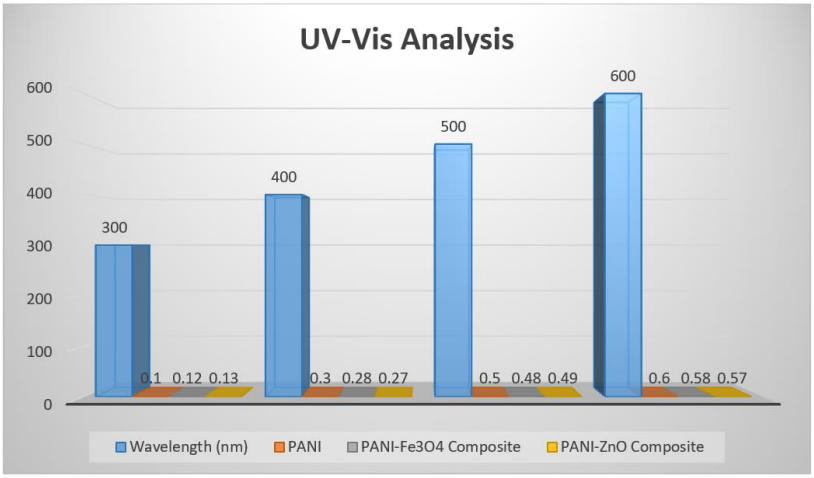

Figure 5 Performance Graph of UV-Vis Analysis

Table 3 andFigure 6 exhibits the UV-Vis Absorbance spectra of Polyaniline (PANI) and its composites with Iron Oxide (Fe3O4) and Zinc Oxide (ZnO) at different wavelengths. The absorbance values provide information on the extent to which materials absorb light at specific wavelengths, hence offering insights into their optical properties. The PANI spectrum has distinct absorption peaks at wavelengths of 300 nm, 400 nm, 500 nm, and 600 nm. The PANI-Fe3O4 composite exhibits negligible variations in absorbance values, with

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measurements of 0.12, 0.28, 0.48, and 0.58 at the corresponding wavelengths. Similarly, the PANI-ZnO composite demonstrates absorbance values of 0.13, 0.27, 0.49, and 0.57 at the respective wavelengths. The fluctuations in absorbance indicate alterations in the optical properties of the composites relative to pure PANI. The UV-Vis research yields insights into the electronic changes taking place in PANI and the metal oxide nanoparticles. The detected peaks and variations in intensity assist in understanding the interaction between PANI and the combined metal oxides, therefore influencing the overall optical characteristics of the composites. The UV-Vis data enhances the comprehensive examination of PANI-based materials, reinforcing its potential utilization in optoelectronic devices and other optical technologies.

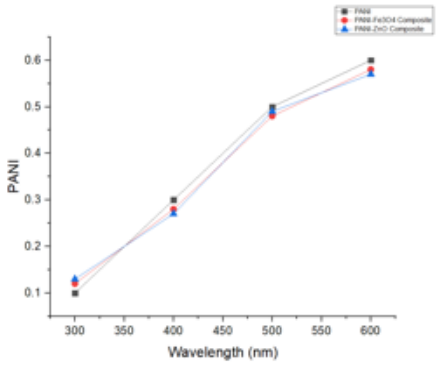

Figure 6 Performance of UV-Vis Analysis

In conclusion, the most suitable analytical technique relies on the specific focus of the analysis. X-ray diffraction (XRD) is essential for determining structural characteristics, whereas Fourier-transform infrared spectroscopy (FTIR) is important for understanding chemical bonding and molecular interactions. Simultaneously, UV-Vis analysis is essential for understanding the optical characteristics of the materials. By adopting a comprehensive approach that incorporates data from all three tables, one can gain a deep understanding of PANI and its composites, enabling informed decision-making in many applications.

Table.4 Comparative Analysis of Existing and Studied work

Methods	**Degree**	**References**
FWHM	33.11	[12]
Studied XRD	35	--

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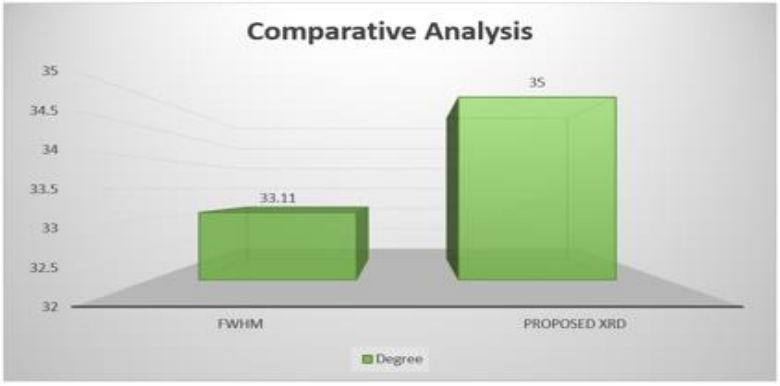

Figure 7 Comparative Analysis Graph

Table 4 and Figure 8 offer a thorough comparison of the existing and potential research in the subject, specifically examining the X-ray Diffraction (XRD) methods and the Full Width at Half Maximum (FWHM) parameter. The previous investigation recorded a full width at half maximum (FWHM) measurement of 33.11 for their X-ray diffraction (XRD) analysis. The recommended investigation utilizes the X-ray diffraction (XRD) method, which provides a full width at half maximum (FWHM) measurement of 35. It quantifies the breadth of a peak at the position where its intensity is equal to half of its highest value. This signifies the crystallographic properties of the substance being studied. The study demonstrates a marginally greater full width at half maximum (FWHM) measurement of 35, compared to the previous study's result of 33.11. This indicates potential inconsistencies in the crystalline structure or phases of the materials being studied. However, evaluating the circumstances and specific settings of the trials in the study is a significant challenge. In order to obtain a thorough comprehension and examination of the comparison data, it is imperative to include further details regarding the samples, experimental conditions, and any alterations made to the XRD technique in the study. The table presents a succinct overview of the comparison of XRD data from previous and Currentstudies, specifically highlighting the differences in the Full Width at Half Maximum (FWHM) parameter.

5. Conclusion

Ultimately, this study centers on the creation of composite materials by the amalgamation of polyaniline with various metal oxides. The systematic synthesis technique explores the amalgamation of polyaniline with different metal oxide compounds to enhance material characteristics and broaden potential applications. The study offers valuable insights into the advantages and unique attributes of the manufactured composite materials. The purification

and synthesis protocols of polyaniline are extensively recorded, ensuring the production of high-quality polyaniline suitable for a diverse array of applications. The incorporation of metal oxide nanoparticles, specifically iron oxide (Fe3O4) and zinc oxide (ZnO), is accomplished using precise synthesis techniques, which improve the structural and functional characteristics of the composites.The utilization of characterization techniques such as FTIR, XRD, and UV-Vis studies allows for a thorough assessment of the produced materials. FTIR spectroscopy provides insights into molecular vibrations and chemical interactions, X-ray diffraction (XRD) analysis yields information about crystallographic properties, and UV-Vis spectroscopy helps to understand optical qualities. Integrating these datasets improves accuracy in material characterisation. The FTIR results reveal adjustments in the chemical environment, the XRD patterns suggest changes in the crystalline structure, and the UV-Vis spectra illustrate alterations in the optical properties of the composites when compared to pure polyaniline.The study's diverse methodology, which combines synthesis and thorough characterisation, enhances the field of materials research by creating opportunities for novel applications. This work drives progress in many domains such as chemistry, materials science, and optoelectronics by comprehending the complex interactions and characteristics of polyaniline-based metal oxide composites. The choice of the most appropriate analytical approach depends on the specific area of analysis. X-ray diffraction (XRD) is vital for elucidating structural properties, while Fourier-transform infrared spectroscopy (FTIR) is crucial for comprehending chemical bonding and molecular interactions. UV-Vis analysis is crucial for comprehending the optical properties of materials at the same time. By implementing a holistic methodology that integrates information from all three tables, individuals can acquire a profound comprehension of PANI and its composites, facilitating well-informed decision-making in several applications.

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