

POLYMER SEMICONDUCTOR POLYMER NANO COMPOSITE PRODUCTION AND CHARACTERIZATION

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ABSTRACT

The focus of this investigation is on polymer semiconductor nanocomposites, namely those that are composed of metal oxides and polyaniline (PANI). The structural, electrical, and dielectric properties of the advanced nanocomposites that are produced as a result of combining PANI with nanoparticles of metal oxides—such as magnesium oxide, zinc oxide, and iron oxide—will be improved. Metal oxide nanoparticles are produced using a combination of in-situ polymerization and a low-temperature combustion process, which results in the creation of nanocomposites. In the process of analyzing the structural features of the materials that were formed, X-ray diffraction (XRD) was utilized to confirm that the metal oxides were crystalline, while PANI was found to be amorphous. Scanning electron microscopy (SEM) was used to examine the morphology, and the results showed that the nanoparticles were equally dispersed throughout the polymer matrix. This was evidence of the presence of nanostructures, which had an average particle size of roughly 35-40 nm. The Fourier transform infrared spectroscopy (FTIR) technique was utilized in order to verify the presence of functional groups and chemical bonds inside the composites. In order to investigate the electrical qualities, the conductivity of direct current (DC) and alternating current (AC) was measured at a variety of frequencies and temperatures. It was discovered through the measurement of the dielectric constant and the dielectric loss across a number of frequencies that the presence of metal oxide nanoparticles had a substantial impact on the dielectric properties of PANI.

Keywords: X-ray diffraction, semiconductor, conducting polymers including polyaniline, scanning electron microscopy

Introduction

Over the past several years, polymer-based semiconductor nanocomposites have attracted a lot of attention because to the exceptional mix of electrical, optical, and mechanical capabilities that they possess. These materials incorporate semiconductor nanoparticles into polymer matrices, which improves their performance in a variety of technical applications. These applications include flexible electronics, sensors, energy storage, and optoelectronic devices. As a result of the synergy that exists between the polymer matrix and semiconductor nanofillers, we are able to achieve adjustable electrical conductivity, increased mechanical stability, and enhanced thermal characteristics. This enables us to consider these materials as viable alternatives to standard inorganic semiconductors. The study into the

synthesis, processing, and characterisation of polymer semiconductor nanocomposites has been further fuelled by the rising need for electronic materials that are lightweight, flexible, and cost-effective.

In situ polymerisation, solution blending, melt mixing, and electrospinning are some of the synthesis strategies that are utilised in the manufacture of these nanocomposites. Each of these approaches offers different benefits in terms of attaining homogenous dispersion of nanofillers inside the polymer matrix. When it comes to defining the overall qualities of the composite, the role of the polymer and nanofiller that you choose to use is quite significant. Because of their intrinsic electrical conductivity and durability, conducting polymers including polyaniline (PANI), polythiophene (PTh), and poly(3,4-ethylenedioxythiophene) (PEDOT) are frequently utilised in a variety of applications. Graphene and carbon nanotubes (CNTs) are two examples of carbon-based nanomaterials that are commonly used to improve charge transport and electrical characteristics. On the other hand, semiconductor nanoparticles such as titanium dioxide (TiO₂) and zinc oxide (ZnO) are also widely used.

In order to gain a knowledge of the structural, morphological, and electrical behaviour of these nanocomposites, conducting characterisation is very necessary. For the purpose of analysing the chemical interactions, crystalline structure, and dispersion of nanoparticles within the polymer matrix, various techniques are utilised. These techniques include Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). Techniques such as four-probe conductivity tests and impedance spectroscopy are utilised in order to evaluate the electrical characteristics of the material. On the other hand, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) are utilised in order to evaluate the thermal stability of the material. There are enormous implications for the next generation of electronics brought about by the discovery of polymer semiconductor nanocomposites. These nanocomposites provide prospects for the creation of wearable devices, flexible solar cells, and high-performance transistors. However, in order to fully realise their potential, it is necessary to overcome issues such as the agglomeration of nanoparticles, the compatibility with polymers, and the stability over long periods of time. The purpose of this research is to investigate the synthesis, processing, and complete characterisation of polymer semiconductor nanocomposites. The findings of this investigation will provide insights into the functional features of these nanocomposites as well as potential applications in contemporary industry.

Objectives

1. To analyze the direct current conductivity of polyaniline and its nanocomposites as a function of temperature.
2. To determine the activation energy of polyaniline and its nanocomposites through temperature-dependent conductivity measurements.

Method

We are able to acquire analytical grade aniline monomer, ammonium persulfate (APS), methanol, acetone, magnesium nitrate hexahydrate, zinc nitrate hexahydrate, and iron nitrate hexahydrate from S D

Fine Chemicals, which is located in Mumbai. In order to produce polyaniline and its nanocomposites, the following chemical components and a variety of other materials were utilized in the synthesis process: conical flasks, beakers, a magnetic stirrer, a funnel, and whatman filter sheets. In order to produce magnesium oxide nanoparticles, magnesium nitrate hexahydrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) was first used, and then an Aloe Vera gel solution was utilized as the fuel. In order to produce zinc oxide nanoparticles, a combination of zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and fuel Aloe Vera gel was utilized. Additionally, the creation of Fe_2O_3 nanoparticles was accomplished through the utilization of Aloe Vera gel solution and iron nitrate hexahydrate [$\text{Fe}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$].

S N	Nano-particles	Inorganic compound	Fuel used
1	Magnesium Oxide(MgO)	Magnesium nitrate hexahydrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$)	Aloe Vera gel solution
2	Zinc Oxide (ZnO)	Zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$)	AloeVera gel solution
3	Iron Oxide (Fe_2O_3)	Iron nitrate hexahydrate ($\text{Fe}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$)	AloeVera gel solution

Result

Measurements of DC conductivity of prepared samples

Our objective was to determine the DC conductivity (σ_{dc}) of the samples by conducting resistivity tests on them. The initial step was the measurement of resistance, which was then followed by the computation of resistivity (ρ). Finally, the dc conductivity was produced by calculating the reciprocal of the resistivity. It was determined that the activation energy of the samples may be evaluated by using the DC conductivity of the materials. The DC conductivity of PANI and its nanocomposites, such as PANI/MgO, PANI/ZnO, and PANI/ Fe_2O_3 , was evaluated using a range of different weight percentages of individual components. Through the use of human labor, the measurement was carried out between 313 and 453 degrees Celsius. A Keithly digital multi-meter with a constant current source was utilized for the measurements, which were performed with the two-probe method. The model number of the multi-meter is 6514. The measurement of resistance as a function of temperature is depicted in the pictorial representation shown in figure 3.19.



Fig3.19: Experimental set-up for DC conductivity measurements

The apparatus used in the experiment consists of a heating furnace, a variable current (variac), a Keithley meter (which supplies a constant current), a sample holder with two electrodes, and other similar components. In order to thoroughly investigate the DC conductivity of polyaniline, it is essential to measure the resistance of the produced PANI product. Following the application of PANI pellets to the sample container, the pellets were then heated in a furnace. The sample is heated in the heating furnace to a temperature of 160 degrees Celsius because the melting point of polyaniline is 180 degrees Celsius. When the temperature hits 160 degrees Celsius, the heating is switched off and the temperature is allowed to cool down. As part of the process of cooling, the reading from a Keithely-6514 electrometer is manually recorded each time the temperature drops by fifty degrees Celsius. On display in Table 3.3 are the typical resistance statistics for Polyaniline (PANI) that have been measured through experimentation.

The same experimental method is also used to evaluate the resistance of pellets that are composed of PANI/MgO nanocomposites with a weight proportion of 10%. The PANI/MgO nanocomposites pellet was placed inside the sample holder, and then the holder was heated in a furnace after the pellet was included. In the case of the nanocomposite sample, a temperature of 160 degrees Celsius was attained. The resistance is measured by a Keithely electrometer, and the reading is recorded each time the temperature drops by fifty degrees Celsius. PANI/MgO nanocomposites are included in Table 3.3, which also contains the typical resistance statistics that were measured through experimentation. The same method is also used to measure the resistance of nanocomposites made of PANI and magnesium oxide at 30 and 50 various weight percentages of magnesium oxide. The measurements are taken as a function of

temperature.

Furthermore, the measuring of resistance in relation to temperature is achieved by employing the same method that was explained before in this paragraph. The data of measured resistance for the other samples of PANI/ZnO and PANI/Fe₂O₃ nanocomposites at 10, 30, and 50 different weight percentages are used to study the DC conductivity and activation energy. This is done utilizing the data.

Table3.3: Resistance measured at different temperature for PANI and PANI/MgO nanocomposites

Sl. No.	Temperature (T ⁰ K)	Resistance(RinKΩ) PANI	Resistance(RinKΩ) PANI/MgOat10wt%
1	323	1.79E-07	2.83E-07
2	328	1.70E-07	2.74E-07
3	333	1.68E-07	2.69E-07
4	338	1.62E-07	2.65E-07
5	343	1.59E-07	2.59E-07
6	348	1.56E-07	2.50E-07
7	353	1.54E-07	2.39E-07
8	358	1.52E-07	2.28E-07
9	363	1.49E-07	2.15E-07
10	368	1.45E-07	2.07E-07
11	373	1.41E-07	1.97E-07
12	378	1.37E-07	1.96E-07
13	383	1.35E-07	1.93E-07
14	388	1.32E-07	1.88E-07
15	393	1.28E-07	1.83E-07
16	398	1.26E-07	1.80E-07
17	403	1.25E-07	1.74E-07
18	408	1.23E-07	1.67E-07
19	413	1.19E-07	1.58E-07
20	418	1.19E-07	1.54E-07

Synthesis of Polyaniline

The synthesis of polyaniline (PANI) was accomplished through the use of the following technique, which is depicted in the flow chart located in 2. In a volume of 100 milliliters of double-distilled water, a mixture consisting of 0.2 millimolar aniline monomer and 0.2 millimolar hydrogen chloride; the ratio is 1:1. The mixed solution was agitated in a beaker in a nice-bath that was set at a temperature of five degrees Celsius until it became clear, as illustrated in Figure 3.1. The combination was then given a drop-by-drop addition of an oxidizer that was a solution of ammonium persulfate with a concentration of 0.4 M. After the combination was mixed, the color of the mixture changed to a dark green. After being stirred continuously for six to eight hours at a temperature of fifty degrees Celsius, the dark green solution was allowed to dry. After a period of twenty-four hours, the solution was filtered through

Whatman filter paper, and the precipitate was extracted. illustrates the outcomes of numerous washes with acetone, which were performed in order to remove any pollutants that were still present in the precipitate that was collected. Following a drying time of twelve hours in air, the precipitate was then placed in a hot-air oven that was adjusted to sixty degrees Celsius. An example of the finished product, a polyaniline yield product.



Fig 1: A photograph for solution mixing using magnetic stirrer

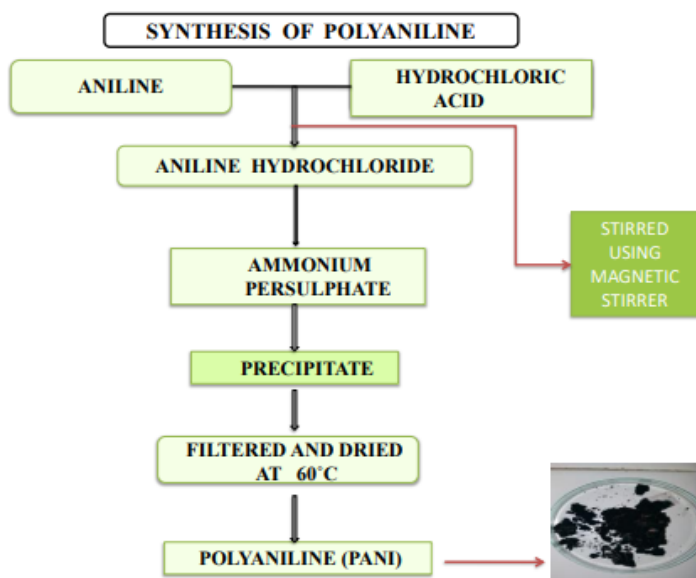


Fig 2: Flowchart for synthesis of PANI

Synthesis of MgO Nanoparticles and Polyaniline Nanocomposites

Preparation of MgO nanoparticles

In accordance with the findings presented in Figure 3.3, the MgO nanoparticles were produced through the utilization of the low temperature combustion technique. A amount of magnesium nitrate hexahydrate $[Mg(NO_3)_2 \cdot 6H_2O]$ of analytical grade was used as the precursor in the production of magnesium oxide nanoparticles. Additionally, an aloe vera gel solution was utilized as the precursor. There were 3.14 grams of magnesium nitrate that were placed inside of a beaker. A gel solution containing twenty milliliters of aloe vera was added to the beaker, and the mixture was agitated for roughly twenty minutes after the addition. After a transparent solution has been formed, it is then heated to a temperature of 500 degrees Celsius in a muffle furnace and allowed to remain there for fifteen minutes so that it can undergo combustion. When the mixture was brought to a boil, the end result was a residue that had a white color to it. For the objectives of characterization and additional PANI doping, the material that was left over was collected. Through the utilization of the Debye-Scherrer formula, the average crystallite size of the magnesium oxide nanoparticles was verified. The X-ray diffraction (XRD) spectra of the strong intensity peak revealed that the particle size was 35 nanometers.



Fig 3: Flow chart for preparation of MgO nanoparticles

Preparation of PANI/MgO nanocomposites

An in-situ polymerization approach was utilized in order to successfully produce nanocomposites consisting of PANI and MgO. The method of creating nanocomposites is depicted in the flow diagram that can be seen in Figure 3.4. A solution consisting of 0.2 M aniline monomer and 0.2 M hydrochloric

acid, both of which are dissolved in 100 ml of double-distilled water, with a ratio of 1:1. The mixture was swirled in a beaker that was placed in an ice bath at a temperature of fifty degrees Celsius until it became clear, as depicted in Figure 3.1. In the following step, ten percent by weight of magnesium oxide nanoparticles were introduced into the aniline hydrochloride sample. The mixture was subjected to a gradual addition of an oxidant solution consisting of 0.4 M ammonium per sulfate until it eventually developed a dark green color. Before allowing the dark green solution to dry, it was heated to a temperature of fifty degrees Celsius and agitated continuously for six to eight hours. After a period of twenty-four hours, the solution was filtered through Whatman filter paper, and the precipitate was extracted. The precipitate that was obtained was washed with acetone on many occasions in order to remove any impurities that were still present. It was first dried in air before being dried in a hot-air oven at a temperature of sixty degrees Celsius for a period of twelve hours. After much effort, it was ultimately possible to generate a PANI yield product that contained MgO nanocomposite.

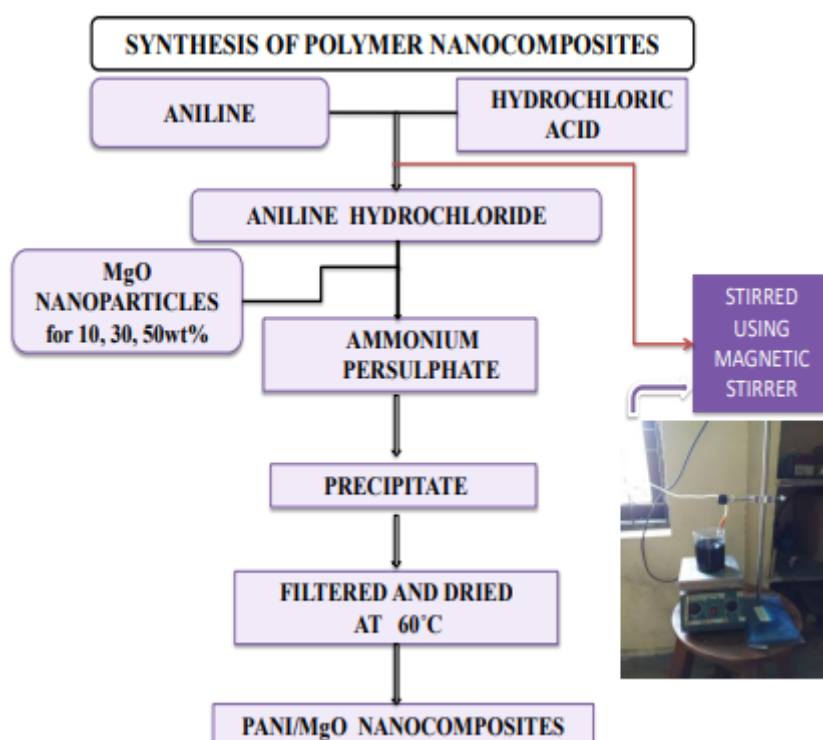


Fig 4: Flowchart for synthesis of PANI/ MgO nanocomposites

In the same manner, the technique was carried out for different percentages of magnesium oxide with PANI nanocomposites, such as thirty and fifty percent by weight. After the powder has been prepared, it is heated to sixty degrees Celsius and then placed in a hot air oven for twelve hours to dry. Pellets made from the powder were used in additional research on capacitance, phase angle, dissipation factor, impedance AC conductivity, and DC conductivity. These investigations were carried out utilizing the powder.

X-ray diffraction (XRD):

The X-ray diffraction spectra for conducting Polymer of Polyaniline, metal oxide nanoparticles of MgO,

ZnO, Fe₂O₃ and nanocomposites of PANI/MgO, PANI/ZnO and PANI/Fe₂O₃ were obtained using the X-ray diffraction (XRD) technique. The source used for the production of X-ray through the experiment are CuK_α ($\lambda = 1.542 \text{ \AA}$) [Rigaku-Ultima IV] with a scan rate of four degree per minute in the range of 10 to 90° angles of 2 θ value. With the analysis of XRD spectra it will be confirmed the nature of the synthesized polyaniline, nanoparticles and polymer nanocomposites. The crystallites size (D) of the synthesized samples was calculated using the Debye Scherer formula given in equation (4.1). Where K is constant = 0.9, β is Full width at half maximum (FWHM) of the spectrum, λ is the wavelength of X-ray source used ($\lambda = 1.542 \text{ \AA}$) and θ is the diffraction angle.

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (\text{\AA})$$

XRD spectra of Polyaniline (PANI)

After being installed on the sample holder, the PANI powder sample is then placed inside the scattering chamber. This serves as the target material. When the sample is scanned at a rate of 4 degrees per minute, the incident X-rays of CuK_α ($\lambda = 1.542 \text{ \AA}$) are able to be detected at an angle of 2 θ . As shown in Figure 4.1, the XRD spectra of the polyaniline polymer are available for evaluation.

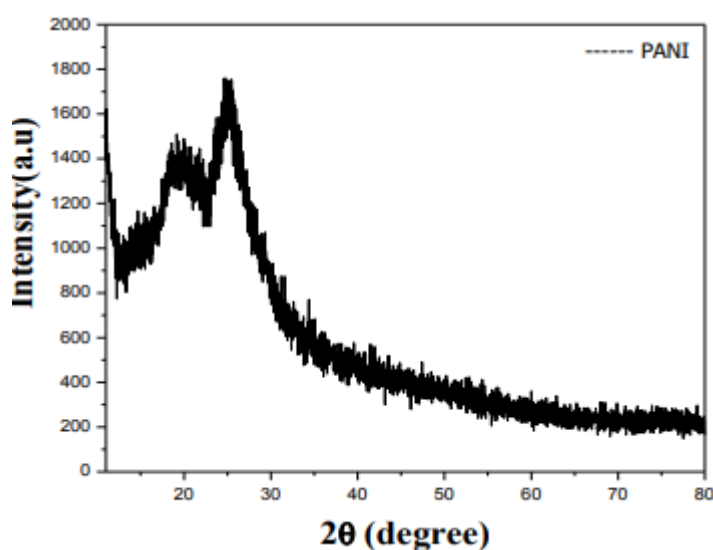


Fig 5 XRD spectra of polymer of polyaniline

Within the X-ray diffraction (XRD) spectrum of PANI, the broad peak, which is the typical peak of PANI, was observed at a 2 θ value of 25.58°. The broad diffused XRD spectra of polyaniline (PANI) demonstrate that it is an amorphous and non-crystalline substance. In order to determine the size of the crystallite, the FWHM (β) value was utilized. The Debye-Scherer formula, which can be found in equation (4.1), was utilized in order to ascertain that the crystallite size of the polyaniline was forty nanometers.

XRD spectra of Magnesium oxide (MgO) nanoparticles

Figure 4.2 depicts the XRD spectrum of the MgO nanoparticles that were created. These nanoparticles were synthesized. Based on the data presented in Figure 4.2, it can be observed that the sample that was

created exhibits distinct diffraction peaks of magnesium oxide nanoparticles at 37 degrees, 43 degrees, 62 degrees, 74 degrees, and 78 degrees 2θ angles. These angles result in miller indices values of (111), (200), (220), (311), and (222) respectively. The diffraction peaks provide a good indication of the crystal structure of the cubic (halide) phase of magnesium oxide represented by JCPDS card number 87-0652. As a result of the very concentrated peaks that were seen at angles of 37 degrees, 43 degrees, 62 degrees, and 74 degrees and 78 degrees, the samples were most likely crystalline [Krishna Moorthy S et al. (2015)]. The determination of the size of crystallites was accomplished by utilizing the values of the total width at half maximum (β) of the strong peaks in the intense spectra. On the basis of the XRD spectrum, the Debye-Scherrer formula was utilized in order to ascertain the specific particle size of the magnesium oxide nanoparticles. After careful analysis, it was discovered that the average size of the crystals was 35 nanometers.

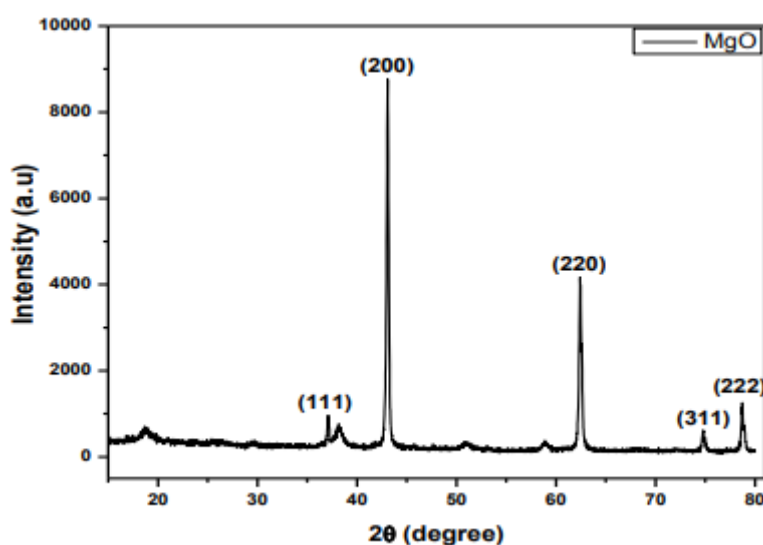


Fig 6 XRD spectra of MgO nanoparticles

XRD spectra of PANI/MgO nanocomposite

The PANI/MgO nanoparticles that have been synthesized are shown in Figure 4.3, which illustrates the XRD spectrum of these nanoparticles. Presented here are the X-ray diffraction spectra of the PANI/MgO nanocomposite. The spectra exhibit the maximum points at an angle $2\theta = 43^\circ$ with plane (200) for a PANI/MgO content of 30%. Additionally, a peak is observed at an angle $2\theta = 62^\circ$ with plane (220) for a PANI/MgO content of 50%. It was determined that the presence of magnesium oxide nanoparticles in a polymer matrix of polyaniline was confirmed by the detection of peaks that are characteristic of these particles. Additionally, the semi-crystalline nature of the PANI/MgO nanocomposites was also seen. In addition, the findings indicate that the MgO nanoparticles covered the surface of the polyaniline. This was evidenced by the fact that the molecular chain affected the crystal structure of the nanoparticles, which led to a reduction in the crystallinity of the nanoparticles. As a result, this demonstrates that the structure of magnesium oxide (MgO) has been maintained and that it is replaced in the polyaniline during the process of polymerization.

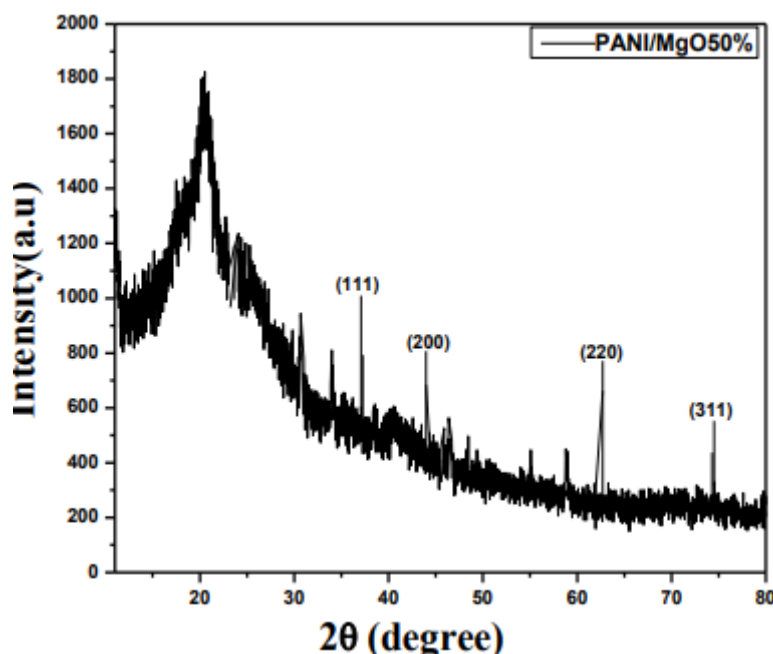


Fig 7 XRD spectra of PANI/MgO nanocomposites

The crystallite size of the PANI/MgO particles did not change after the doping process was completed. By applying the Debye-Scherrer formula, we were able to determine that the typical size of the crystals was 32 nanometers by using the formula.

Conclusion

Advanced electrical and optoelectronic applications should benefit greatly from polymer semiconductor nanocomposites, which have shown great promise in synthesis and characterisation. Nanocomposites made of polymer matrices and semiconductor nanoparticles have great potential as next-generation transistors, sensors, energy storage devices, and flexible electronics due to their high electrical conductivity, increased mechanical strength, and exceptional thermal stability. In order to maximise performance and guarantee uniform dispersion of nanofillers within the polymer matrix, research into several synthesis methods, such as melt mixing, solution blending, and in situ polymerisation, has yielded important insights. In addition, electrical conductivity tests, scanning electron microscopy (SEM), transmission electron microscopy (FTIR), and XRD have all contributed to a better understanding of the basic interactions that impact the material characteristics, such as those between polymer chains and semiconductor nanoparticles. The benefits of these nanocomposites seem promising, but there are still various obstacles to overcome in order to produce them on a big scale and ensure their stability over the long term. Innovative techniques, such surface functionalisation of nanoparticles and enhanced processing technologies, are needed to comprehensively address issues like nanoparticle aggregation, phase separation, and polymer compatibility. In order for these composites to withstand the extreme conditions encountered by contemporary electrical devices, more study is needed to improve their mechanical flexibility and environmental stability.

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