A review on synthesis and characterization of Polyaniline (PANI) metal oxide nano composites

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Abstract: *Polyaniline, commonly abbreviated as PANI or Pani—also known as aniline black— was discovered for the first time by Ferdinand Runge in 1834. PANI metal oxide composites can be synthesized using both chemical and electrochemical oxidative polymerization in acidic media. The most widely used initiators or oxidants for the chemical polymerization of aniline. Synthesised PANI composites were subjected to XRD, FTIR to understand the structural modifications. UV-Visible studies show the optical properties and dielectric studies shows the electrical conductivity variations with dopants.*

Index Terms **-** PANI, conducting polymer, nanocomposites, XRD-spectra

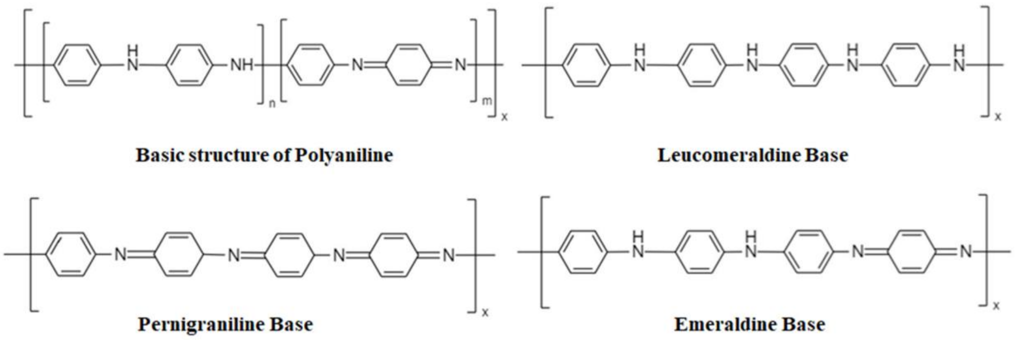
I. INTRODUCTION

For decades, the world of science and research has been captivated by the extraordinary electrical and electronic properties of conducting polymers. These wonder materials, also known as intrinsically conducting polymers (ICPs)**,** defy the expectations set by traditional insulators like plastics. Unlike their insulating counterparts, ICPs boast the remarkable ability to conduct electricity, behaving akin to metals or semiconductors [1]. This enhances potential applications across diverse fields. The conductivity of conducting polymers is a spectrum, spanning the range from semiconductors to metals. This depends on the specific polymer and its doping level. Doping, a process involving the introduction of electron donors or acceptors into the polymer chain, serves as a magic knob for fine-tuning the electrical, optical, and even mechanical properties of these materials. The processing possibilities for conducting polymers are just as diverse as their properties. From films and fibres to tubes, these versatile materials can be crafted into various forms using techniques like chemical synthesis, electrochemical polymerization, and spin coating [2-3]. This opens doors for their integration into a wide range of applications, especially in the realm of flexible electronics**.** Among the plethora of ICPs, Polyacetylene (PA), Polypyrrole (PPy), Polythiophene (PTh), and Polyaniline (PANI) are the names that frequently grace research papers and hold immense promise for the future. They can revolutionize area such as energy storage**,** solar cells, microelectronic devices, sensors, and even optoelectronic gadgets**.** Polyaniline (PANI) **a** semi stiff rod polymer has been captivating researchers since the 1980s with its exceptional electrical conductivity and impressive mechanical properties [4-5]. When doped with acids or other agents, its conductive property can be used in electronic applications. Depending on the chosen dopant and oxidation state, its conductivity and even color can be adjusted making it ready to adapt to diverse needs. Compared to its fellow ICPs, PANI boasts an extra layer of allure – its resilience. It exhibits admirable resistance to environmental factors like temperature and light, making it a practical choice for real-world applications [6-7]. PANI can be made readily available by diverse chemical and electrochemical synthesis methods. The unique combination of high conductivity, tuneable properties, and ease of processing makes conducting polymers, and especially PANI, a beacon of hope for the future. From flexible electronics that seamlessly blend with our lives to the development of efficient energy storage solutions, these materials hold the key to unlocking a world of possibilities.

II. Literature review

In recent decades, conducting polymers have gained significant attention in academic and scientific domains due to their exceptional and distinct electrical and electronic characteristics [8]. Polyaniline, commonly abbreviated as PANI or Pani—also known as aniline black— was discovered for the first time by Ferdinand Runge in 1834 from the initial oxidation of aniline. Later, in 1862, Henry Letherby achieved its first electrochemical polymerization. Since then, it has been applied in a variety of colored materials and dyes. Recently, PANI has gained much attention of the scientific community due to the rediscovery of its conductive properties in the 1980s. Its structure consists of a succession of aromatic rings of the benzene diamine and/or quinone di-imine types, bridged by a nitrogen heteroatom. Conducting polymers are a family of organic polymers with electrical conductivity, commonly referred to as intrinsically conducting polymers (ICPs). ICPs are different from conventional insulators like plastics in that they have special characteristics that let them act like semiconductors or metals. This amazing skill offers up a plethora of possible uses in several industries. Depending on the polymer and degree of doping, conducting polymers can have conductivities ranging from those of semiconductors to those of metals. Doping, which is the process of adding electron donors or acceptors to the polymer chain, makes it simple to adjust the electrical, optical, and mechanical characteristics of conducting polymers. Conducting polymers can be processed by several methods, including chemical synthesis, electrochemical polymerization, and spin coating, into different shapes, such as films, fibres, and tubes. Conducting polymers are lightweight and flexible in comparison to conventional conductors like metals, which makes them appealing for use in flexible electronics applications. Polyacetylene (PA), Polypyrrole (PPy), Polythiophene (PTh), and Polyaniline (PANI) are a few of the most widely used ICPs. These CPs belong to the family of organic materials that have potential uses in solar cells [9], microelectronic devices [10], piezoelectric actuators [11], sensors [12], energy storage [7, 13], and optoelectronic devices [14]. Recently, many articles [15–25] described how to produce conducting polymer nanocomposite that incorporates metal oxide nanostructures, such as ferrates, zinc oxide, MnO_2, SnO_2, TiO_2, and others. The conductivity of the nanocomposites may be increased by the CPs acting as conductive supports between polymer chains.

A conducting polymer and organic semiconductor belonging to the family of semi-flexible rod polymers is polyaniline (PANI). The compound's mechanical and electrical conductivity qualities have drawn attention since the 1980s. Among conducting polymers that have been investigated the most is polyaniline. PANI can be used in electronic applications because of its conductivity, which can rival that of metals when doped with acids or other dopants. PANI's conductivity and color may be changed based on the dopant and oxidation state, providing flexibility for a range of applications. PANI's practical application is enhanced by its strong resistance to environmental elements such as light and temperature, as compared to other conducting polymers. Several chemical and electrochemical techniques may be used to produce PANI, which makes it easily accessible and highly producible. Because of their exceptional thermal stability, easy synthesis, unique conduction mechanism, and high conductivity, this has attracted the attention of many researchers [12,13,15]. The following factors make PANI the most conductive polymer: simple production, inexpensive, high conductivity, superior wave absorption, unique doping method, and electrochemical performance [26].



Basic structure of Polyaniline

Leucomeraldine Base

Pernigraniline Base

Emeraldine Base

Figure 3. Different configurations of PANI structures.

In this paper, the researcher made an attempt to present overview of PANI synthesis, characterization and potential applications through previously published works**.**

III. Results and Discussion

3.1 Materials and Method of synthesis

Polyaniline (PANI) nanocomposites offer exciting possibilities for various applications due to their unique combination of conducting polymer properties and nanoscale features. All high purity analytical grade chemicals are employed for synthesis. Some of the major preparation methods;

3.1.1 Chemical Oxidative Polymerization

In-situ polymerization: This common method involves adding aniline monomer to a solution containing an oxidant (e.g., ammonium persulfate) and dopant (e.g., camphor sulfonic acid) in the presence of pre-synthesized nanomaterials like carbon nanotubes, graphene, or metal oxides. The nanomaterials act as templates for PANI growth, leading to uniform dispersion and enhanced properties.Interfacial polymerization: Here, two immiscible phases containing aniline and oxidant/dopant are brought into contact, leading to PANI formation at the interface. This allows for controlled morphology and surface functionality of the nanocomposite [27,33].

3.1.2 Electrochemical Polymerization

Template-assisted electro polymerization: This method uses pre-patterned conductive substrates like gold electrodes to direct PANI growth onto specific areas. Nanomaterials can be incorporated into the electrolyte solution for composite formation.

3.1.3 In-situ solvothermal Synthesis

The in-situ polymerization technique is a versatile and powerful approach for preparing polymer nanocomposites (PNCs) with well-dispersed and strongly-bonded nanofillers. In-situ polymerization involves simultaneous polymerization of the monomer and dispersion of the nanofiller within the reaction medium. This leads to the nanofiller particles being directly incorporated into the growing polymer chains, resulting in Enhanced interfacial adhesion: Strong covalent or ionic bonds form between the polymer matrix and the nanofiller surface, leading to superior mechanical, thermal, and barrier properties. The nanofiller gets trapped within the polymer network during its formation, minimizing agglomeration and ensuring uniform distribution throughout the material increasing dispersion [34].

3.1.4 Microwave-assisted Synthesis

Microwave irradiation can significantly accelerate PANI polymerization and improve its uniformity and crystallinity. This method offers faster reaction times and energy efficiency compared to conventional heating.

3.2 XRD Analysis

XRD studies of PANI composites shows incorporation of metal oxides into the matrix with definite peaks leading the structural modifications which changes crystalline imperfections to improve the electrical conductivity. Some peaks in the XRD patterns of the nanocomposites are shifted and intensified compared to PANI, suggesting interaction between PANI and ZnO. The increased intensity of the peaks in the XRD patterns of the nanocomposites with increasing ZnO content indicates hydrogen bonding formation between PANI and ZnO. The XRD analysis reveals that the presence of TiO2 significantly impacts the crystallinity of PANI in the composite while having minimal influence on TiO2 itself. This suggests a strong interaction between PANI and TiO2 at the molecular level, restricting PANI's crystal growth. The X-ray diffraction pattern's spreading of peaks is often associated with decreasing silver particle sizes. When the sample is created via oxidation with hydrogen peroxide without photolysis, the particles have equal diameters [27-31].

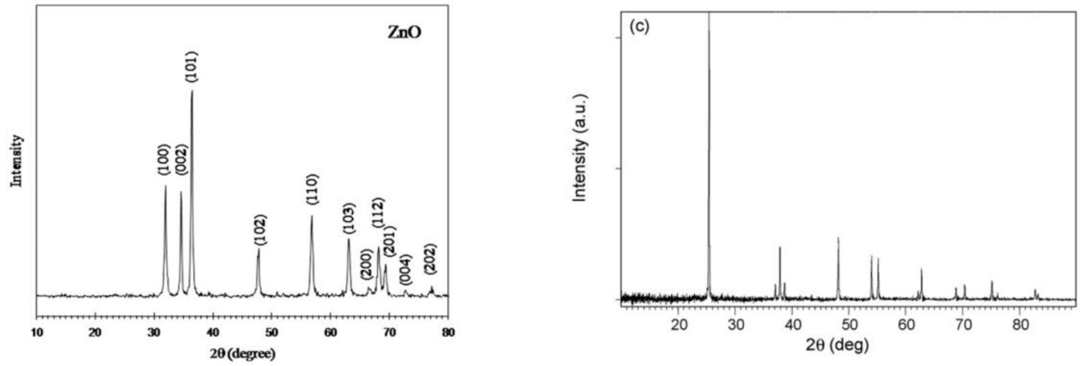


Fig. 1 XRD pattern of ZnO nanorods.

Fig. 3. XRD pattern of (a) TiO_2 (b) PANI and (c) PANI–TiO_2 composite.

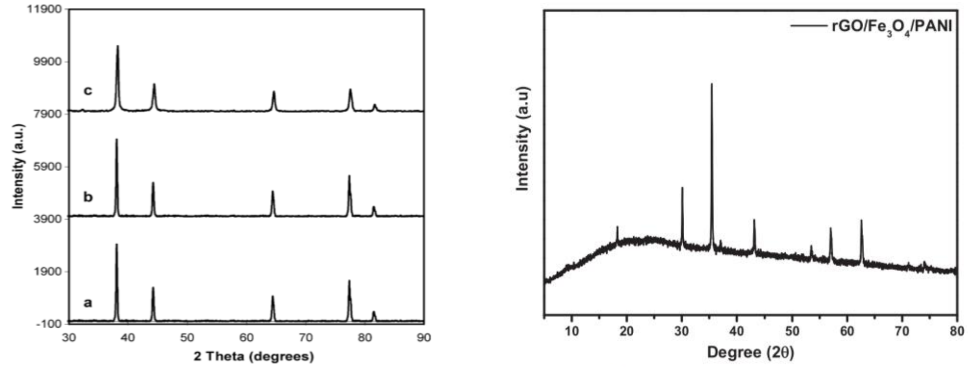


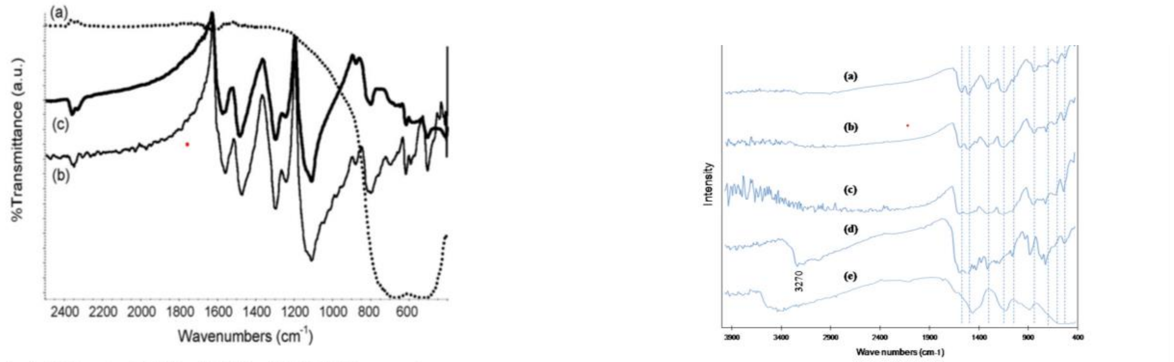
Fig. 7. XRD of (a) Ag–Pani nanocomposite powder prepared at 365 nm

F :RD spectrum of rGO/Fe_3O_4/PANI composite.

Figure-2: XRD spectra of PANI composites

3.3. FTIR Analysis

Fourier transformed IR spectra of samples reveals the effect metal oxides on molecular structures. It is observed change in the vibrational frequency and slight variations in the intensity of vibrational bands. The peak shifts likely alter the electron densities of PANI chains, potentially strengthening C-N and C-C bonds but weakening N-H bonds. It is observed that in some cases increase in concentration of metal oxides filler changes the molecular structural rearrangement and implying uniform distribution and reduced agglomeration in the composites.. The peak at 1722 cm−1 in the rGO/Fe3O4 composite nearly vanishes, and the oxygen functional group intensities significantly decrease, demonstrating the effective reduction of GO by aniline. This suggests that the presence of metal oxide molecules during polymerization restricts PANI's crystal growth. [27-32].



ig. 4. FTIR spectra of (a) TiO_2, (b) PANI and (c) PANI–TiO_2 composite.

FTIR pattern of (a) PANI, (b)–(d) PANI–ZnO 1 wt%, 2 wt% and 4 wt% nanocomposites and (e) ZnO nanorods.

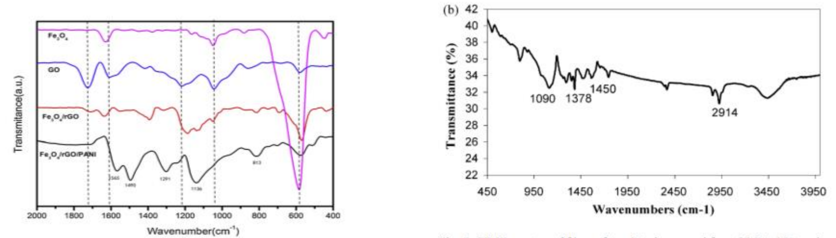


Fig. 2. The FT-IR curves of the pure Fe_3O_4, GO, rGO/Fe_3O_4 and rGO/Fe_3O_4/PANI composites.

Fig. 5. FT-IR spectra of films of Ag–PAni prepared from 2 M $AgNO_3$ solution by (a) 365 nm; (b) 254 nm illumination.

Figure-3: FTIR spectra of PANI composites

3.4. UV-Visible Studies

UV-Visible studies of synthesised PANl composites show the changes in absorbance intensity depending on the concentration & on nature metal oxide. UV-vis spectroscopy was used to analyse the optical properties of PANI and PANI- metal oxide nanocomposites. PANI shows two characteristic absorption peaks at 336 nm and 628 nm, associated with different electronic transitions in its structure. Adding metal oxide nano fillers to PANI showed slight shifts and changes in the intensity of these peaks. ZnO nanorods subtly modify the optical properties of PANI by interacting with specific molecular structures. The extent of these changes depends on the amount of ZnO incorporated. UV-vis spectroscopy is a useful tool for probing the interactions between PANI and ZnO in nanocomposites. Even with increases in silver nano particles changes the absorbance intensity. Similar observation reported to TiO_3 & rGO/Fe_2O_3 composites [27-33].

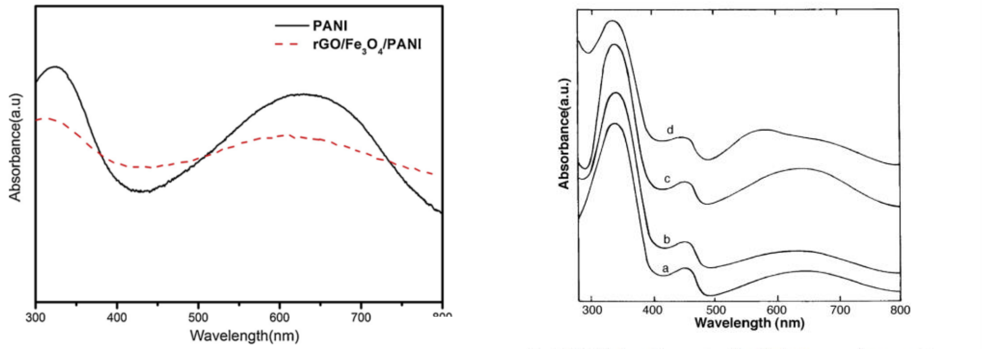


Fig. 4. UV–vis spectra of the PANI and rGO/Fe_3O_4/PANI c

composite (Fig. 4). The PANI displays two characteristic bands at about

Fig. 1. UV–Vis absorption spectra of Ag–PAni nanocomposite prepared by photolysis at 365 nm illumination (re-dissolved solution in DMSO) (a–d; 0.5, 1, 1.5 and 2 M silver nitrate solution used for reaction).

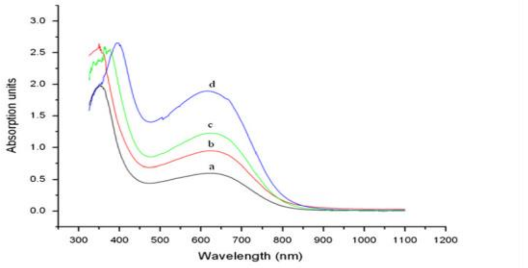


Fig. 8 UV–vis spectra of (a) PANI, (b) PANI–ZnO 1 wt% nanocomposite, (c) PANI–ZnO 2 wt% nanocomposite and (d) PANI–ZnO 4 wt% nanocomposite.

Figure-4: UV-Visible spectra of PANI composites

3.5. Electrical conductivity

The prepared PANI composites were subjected to electrical conductivity studies showing the changes in electrical properties depending on concentration and nature of metal composites. Pure PANI and ZnO nanorods have relatively low conductivities (0.025 S/cm and 0.016 S/cm, respectively). Adding ZnO nanorods to PANI (PANI-ZnO nanocomposites) significantly reduces the overall electrical conductivity (e.g., 0.0068 S/cm for 1 wt.% ZnO). This decrease is attributed to two main factors: Adsorption of PANI's -NH group to the ZnO surface, forming bonds that hinder charge transport [29,35-36]. Lower relative content of conductive PANI due to increasing ZnO concentration. These results are supported by other spectroscopic (TGA, XRD, FTIR) data confirming the interaction between PANI and ZnO. Below table showes Electrical conductivity of PANI, ZnO nanorods and PANI–ZnO nanocomposites. The electrical conductivity analysis reveals that incorporating Ag2O into the PANI/rGO network negatively impacts conductivity. This is expected due to the lower conductivity of Ag2O compared to the PANI/rGO network [37-39]. However, the presence of Ag2O still contributes to the current transport through the nanocomposite, as evidenced by the Ohmic behavior and dependence of conductance on Ag2O content. The AC electrical conductivity and dielectric properties of PANI/Ce–TiO2 nanocomposites with varying Ce–TiO2 shows that incorporating Ce–TiO2 nanoparticles into PANI can significantly improve its electrical conductivity and dielectric properties up to a certain concentration. This finding holds potential for applications requiring high-performance conducting polymers [40-47].

4. Conclusion

This research work delves into the exciting realm of polyaniline (PANI) nanocomposites, highlighting their remarkable potential for various technological advancements. PANI, a conducting polymer renowned for its exceptional electrical and optical properties, can be further enhanced by incorporating a plethora of nanomaterials, leading to the creation of tailored functionalities for specific applications. PANI boasts conductivities comparable to metals when doped, making it a versatile material for electronic devices, sensors, and energy storage systems. Doping and nanocomposite formation offer exquisite control over PANI's conductivity, optical behaviour, and thermal stability, enabling its customization for diverse applications. PANI can be synthesized through various methods, including chemical and electrochemical techniques, providing flexibility and scalability for production. The exploration of PANI nanocomposites with other nanomaterials, such as graphene oxide, metal oxides, and carbon nanotubes, opens up boundless possibilities for future research. Tailoring the interface between PANI and these nanomaterials, along with further optimization of synthesis processes, will pave the way for next-generation materials with unprecedented functionalities. Overall, PANI nanocomposites offer a transformative platform for advancing technologies in diverse fields, from energy harvesting and conversion to bioelectronics and environmental remediation. Their remarkable versatility and tunability position them as key players in shaping the future of materials science and engineering.

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