



SYNTHESIS, CHARACTERIZATION, AND APPLICATIONS OF ZNO–GRAPHENE NANOCOMPOSITES

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ABSTRACT

The synthesis, characterisation, uses, and prospects for ZnO–Graphene nanocomposites in the future offer a constantly evolving field of scientific inquiry and technological advancement. This abstract highlight important elements that define the storey of these cutting-edge materials, thereby capturing the spirit of this complex journey.

The production of ZnO–Graphene nanocomposites is characterised by an ongoing pursuit of accuracy and consistency. To accomplish successful integration, researchers have used a variety of synthetic processes, such as sol-gel methods and chemical vapour deposition. A primary goal is to optimise these techniques in order to guarantee consistency in the properties of the nanocomposites and improve scalability. The synthesis procedure is the cornerstone upon which ZnO–Graphene nanocomposites' special qualities and capabilities are constructed.

When it comes to deciphering the complex structural features of ZnO–Graphene nanocomposites, characterization procedures are essential. Together, Fourier Transform Infrared Spectroscopy (FTIR), Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), and X-ray Diffraction (XRD) provide a thorough understanding of the composition, shape, and internal structure of the nanocomposites. Researchers find these techniques to be quite useful since they offer valuable insights that help fine-tune the properties of nanocomposite materials for certain applications.

ZnO-Graphene nanocomposites have a wide range of applications, which demonstrate their adaptability and versatility. These nanocomposites have remarkable photocatalytic qualities that aid in the breakdown of contaminants in the air and water during environmental restoration. The special combination of graphene's conductivity and ZnO's semiconducting qualities makes for extremely sensitive and selective sensors in sensing technologies. The energy storage capacity of



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the nanocomposites is greatly increased, improving the efficiency of batteries and supercapacitors. They boost solar cell performance in photovoltaics, and their biocompatibility is utilised in biomedical applications for imaging and medication delivery.

ZnO-Graphene nanocomposites have an exciting trajectory of innovation ahead of them. Scientists are ready to further refine synthesis techniques such that they are repeatable and scalable. At the forefront of research are improved nanocomposite qualities designed for particular uses. The next stage of these materials' evolution will be defined by innovative uses, integration with cutting-edge technologies, and a dedication to sustainable methods.

INTRODUCTION

In the field of nanotechnology, the amalgamation of discrete elements to generate nanocomposites has created opportunities for the creation of sophisticated materials possessing innovative characteristics and improved capabilities. Of all the pairings, the one that works best is zinc oxide (ZnO) and graphene. This combination has drawn a lot of attention recently. In order to better understand this composite material and investigate its potential across a range of technological sectors, this research study explores the synthesis, characterisation, and applications of ZnO–Graphene nanocomposites. The application of nanocomposites has emerged as a fundamental aspect of materials science and engineering because of their remarkable characteristics, which stem from the combined actions of their constituent parts. When combined, the different properties of ZnO, a broad-bandgap semiconductor, and graphene, a single sheet of carbon atoms organised in a hexagonal lattice, produce a novel composite material with a wide range of uses.

Due to its characteristics, which closely resemble those of the photocatalyst required for photo-degradation, zinc oxide (ZnO) has demonstrated significant potential recently. These characteristics include reasonable cost, strong oxidation extent, high electro-chemical coupling coefficient, exceptional photosensitivity, bio consistency, high chemical and mechanical stability, exceptional pyroelectric and piezoelectric characteristics, broad range of radiation absorption, etc. ZnO nanostructures, which comprise quantum dots arrays, elongated arrays, planar arrays, and ordered structures, can be categorized as zero-dimensional (0D), one-dimensional (1D), two-dimensional (2D), and three-dimensional (3D). For a number of years, ZnO has contributed to an increase in the quantity of articles about photocatalysis. However, since 2010, there has been a noticeable increase in interest in the use of ZnO–graphene Nano composites in various photocatalytic applications.

This work focuses on the synthesis of ZnO–graphene composites, going into great detail into the variables and methods that affect the final product's qualities. A thorough explanation of the methods used is provided, along with some noteworthy outcomes using ZnO–graphene nanocomposites. Lastly, under UV and visible light conditions, the photocatalytic activity of ZnO-GO or ZnO-rGO in the degradation of contaminants is discussed.

These characteristics have made graphene a material of interest in industries including energy storage, electronics, and sensor technology. The difficulty, though, is getting beyond graphene's drawbacks, which include its propensity to agglomerate and the requirement for carefully managed integration into composite materials. ZnO and graphene combine to overcome the drawbacks of each material alone while maximising their complementary properties. When combined with ZnO's semiconducting capabilities, graphene's distinct electrical, structural, and chemical features produce a nanocomposite that performs better than either of its constituent parts alone.

Significance - ZnO-Graphene nanocomposites are important because they have the potential to transform a wide range of technological applications. Through the integration of ZnO's semiconducting characteristics with graphene's remarkable conductivity, scientists hope to develop materials that are superior in biomedical applications, energy storage, photocatalysis, and sensing. To fully realise the promise of these nanocomposites, careful synthesis and in-depth characterization are essential.

Objectives -

1. **Synthesis Exploration:** Examine and contrast different ways of synthesising ZnO–Graphene nanocomposites and analyse how they affect the distribution, size, and shape of ZnO nanoparticles on graphene sheets.
2. **Characterization Techniques:** Employ sophisticated characterization methods to give a thorough grasp of the structural, morphological, and chemical characteristics of the nanocomposites, such as X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), and Raman spectroscopy.
3. **Applications Assessment:** Examine the uses of ZnO–Graphene nanocomposites in areas like biomedical applications, energy storage, photovoltaics, sensing, and photocatalysis, and assess their capabilities and possible benefits over current materials.
4. **Future Perspectives:** Examine the present status of research and suggest avenues for future development, with a focus on improving the characteristics of nanocomposite materials, streamlining synthesis processes, and investigating novel applications in developing technologies.

To sum up, the combination of ZnO and graphene in nanocomposites provides a promising path toward the development of materials with customised characteristics and a wide range of uses. The goal of this research is to add to the expanding body of knowledge in this area by illuminating the synthesis, properties, and possible uses of ZnO–Graphene nanocomposites. By thoroughly investigating these facets, our goal is to open the door for the creation of novel materials capable of meeting the demands of today's modern environments.

SYNTHESIS METHODS

2.1. Synthesis Methods:

In the pursuit of creating ZnO–Graphene nanocomposites, the selection of an appropriate synthesis method plays a crucial role in determining the overall properties and performance of the resulting material. This section provides an overview of common synthesis methods employed in the creation of these nanocomposites and conducts a comparative analysis to highlight the strengths and limitations of each approach.

Materials:

Zincite is a mineral that contains Zinc oxide and from that we can get it through various chemical methods. As Zinc is a transition metal so it can react with both acids and bases and behave in acidic or basic way to get oxidised or to oxidise other material.

All chemicals used in these methods were of analytical grade and used without further purification. All chemicals were purchased from various industries from India and were provided to me by the University itself. Distilled water was used throughout in all the experiments. Many experimental methods were used to synthesize ZnO by using various materials. Some of the materials we used as precursor or as substrate are:

- i. Zinc sulphate (ZnSO_4)
- ii. Sodium hydroxide (NaOH)
- iii. Zinc acetate ($\text{Zn}(\text{CH}_3\text{CO}_2)_2 \cdot 2\text{H}_2\text{O}$)
- iv. PVA (Poly vinyl chloride)
- v. Hydrogen peroxide (H_2O_2)
- vi. Ethanol ($\text{C}_2\text{H}_5\text{OH}$)
- vii. Diethylene glycol ($(\text{HOCH}_2\text{CH}_2)_2\text{O}$)

Zinc acetate and Zinc sulphate were use as precursors in different methods, and NaOH was used as an inorganic alkali that worked as a reducing agent. In Sol-gel technique, PVA was used as precursor that acted as surface modificant. It has been reported that ZnO nanoparticles synthesized by using zinc precursors with either base or solvent for the combination of OH^- ions to produce zinc hydroxide moieties produces ZnO nanoparticles via dehydration. H_2O_2 helps to produce ZnO as active surface oxygen species, as it converts ZnO into zinc peroxide translucent material.

Methods:

1) Preparation of ZnO by using Zinc sulphate (ZnSO_4):

NaOH was added slowly dropwise to the aqueous solution of Zinc sulphate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) in a molar ratio of 1:2. It was stirred vigorously and was kept like that for 18 hours approximately until we get the white coloured ppt. Afterwards it was filtered in centrifugal machine and was washed

with distilled water for 4-5 times. Then by using muffle furnace at 100o C we dried it until we got the fine powder. That fine powder was calcined at different temperatures such as 300°C, 500°C, 700°C, and 900°C and then the extracted material was stored for later use.

2) Preparation of ZnO by using Zinc acetate (ZnOOCCH3):

We took 1 mole of Zinc acetate (ZnOOCCH3) and added 2 mole of NaOH into ZnOOCCH3 dropwise and stilled vigorously that happened to be a white slurry product. Then it got stirred for 18 hours and we got the white ppt. Then it was filtered in centrifugal machine and was washed with distilled water. Afterwards, by using muffle furnace at 400o C it got calcined. After drying it degrades the white powder like material to dark ash colour.

3) Preparation of ZnO by using PVA (Polyvinyl alcohol):

To prepare ZnO nanoparticle 2 ml of PVA solution (0.1 %) was added to 1 ml of Zinc sulphate (ZnSO₄. H₂O) solution. Then 2 mole of NaOH was added to it dropwise very slowly and was stirred for 18 hours. During this stirring foam like material starts to form and it get turns into white precipitate. This white ppt was filtered with the help of centrifugal machine and then washed with distilled water. Afterwards it was dried in the muffle furnace at 100o C for 2 hours and then in it was grounded to a fine powder and got calcined at 400o C.

2.2. Comparative Analysis:

To evaluate the efficacy of each synthesis method, a comparative analysis is essential. Factors such as cost, scalability, reproducibility, and the ability to control nanoparticle size and distribution should be considered. Additionally, the impact on the structural integrity of graphene during synthesis is a critical parameter.

Method	ZnSO ₄	ZnOOCCH ₃	PVA
Starting Material	Zinc sulphate (ZnSO ₄ .7H ₂ O)	Zinc acetate (ZnOOCCH ₃)	Zinc sulphate (ZnSO ₄ .H ₂ O) + PVA solution
Precipitation Agent	NaOH	NaOH	NaOH
Stirring Time	18 hours	18 hours	18 hours
Calcination Temperature	300°C, 500°C, 700°C, 900°C	400°C	400°C

Notes	Simple and cost-effective. Offers control over particle size through calcination temperature.	Easy preparation. Results in dark ash colour powder.	Uses a stabilizing agent (PVA) to control particle size and prevent agglomeration. Produces a white powder.
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Table – 1 Comparative Analysis of various synthesis methods

Additional Comparison Points:

Yield: The yield of ZnO can vary depending on the method used. The precipitation method (1) generally offers the highest yield.

Purity: All three methods can produce high purity ZnO. However, the calcination temperature needs to be controlled carefully to avoid impurities.

Particle size and morphology: The particle size and morphology of ZnO can be controlled by the preparation method and calcination temperature. The use of PVA (method 3) can help in controlling particle size and preventing agglomeration.

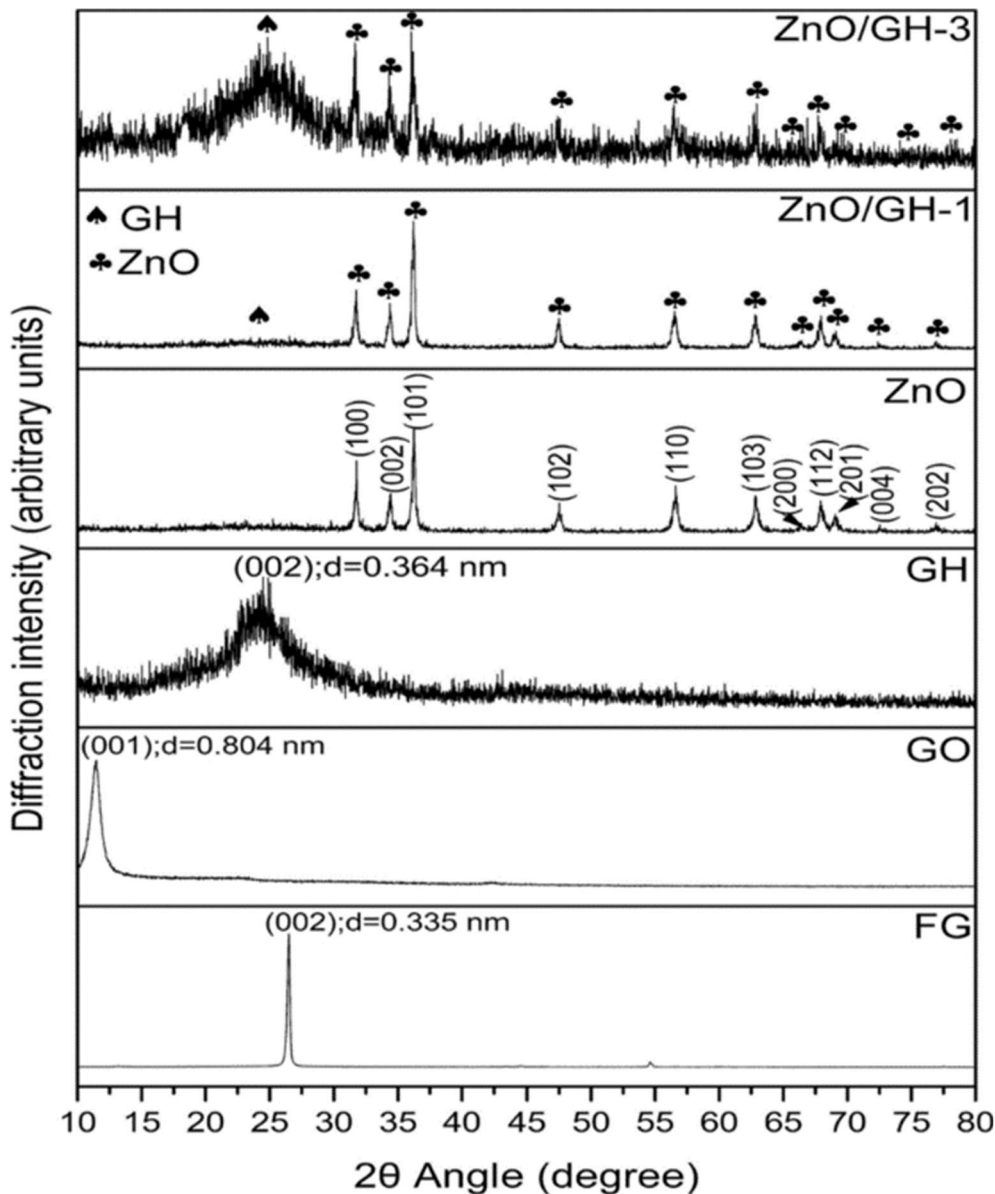
Cost: The cost of the method depends on the starting materials and the equipment used. The ZnSO₄ method (1) is generally the most cost-effective.

The best method for preparing ZnO depends on the desired properties of the final product, such as particle size, purity, and yield. The ZnSO₄ precipitation method (1) is a good general-purpose method that is simple, cost-effective, and offers control over particle size through calcination temperature. The ZnOOCCH₃ method (2) is easier to prepare but results in a dark ash colour powder. The PVA method (3) uses a stabilizing agent to control particle size and prevent agglomeration, which can be beneficial for certain applications.

show up as peaks in the XRD pattern. These peaks, which are specific to each crystalline material, reveal details on the orientation of the nanoparticles inside the composite as well as the phase composition and crystal structure.

XRD Analysis of ZnO-Graphene Nanocomposites

The diffused basal plane (002) peak of graphene and the hexagonal wurtzite structure of ZnO are usually represented by peaks in the XRD patterns of ZnO-graphene nanocomposites. ZnO peaks indicate that ZnO nanoparticles have been successfully incorporated into the graphene matrix. The size and strain of the crystallites within the ZnO nanoparticles can be ascertained by analysing the intensity and width of these peaks. Furthermore, other crystalline phases in the nanocomposite, such as impurities or secondary phases created during production, can also be detected by XRD. Moreover, the degree of exfoliation and interlayer spacing within the graphene sheets can be inferred from the relative strength and position of the graphene peak.



Graph -1 XRD patterns of FG, GO, GH, ZnO, and ZnO/graphene nanocomposites.

Advantages & Limitations of XRD for Characterizing ZnO-Graphene Nanocomposites

The relative abundance of the various phases within the composite can be ascertained by measuring the intensity of the diffracted peaks. X-ray diffraction (XRD) can be used to investigate different facets of the crystal structure, including strain, phase composition, and crystallite size. XRD is unable to reveal details about the nanocomposite's amorphous elements, such as the surface functional groups on the graphene sheets. For XRD analysis to be successful, the sample needs to be crystalline.

In general, XRD is an essential technique for comprehending ZnO-graphene nanocomposites' structural characteristics. It is a crucial approach for researchers creating and refining these promising materials for a range of applications since it can give quantitative information about the orientation, phase composition, and crystal structure of nanoparticles.

Phase Purity

When just the desired phases are present in the nanocomposite, it is referred to as phase purity. Regarding ZnO-graphene, the phases that are wanted are:

- ZnO: Due to its diverse applications, ZnO's hexagonal wurtzite structure is crucial.
- Graphene: For its conductivity and other characteristics, well-defined graphene sheets with few imperfections are essential.

The performance of the nanocomposite might be greatly impacted by the presence of impurities or undesirable phases. By serving as scattering centres, impurities can lower conductivity and other capabilities. Furthermore, they may alter the stability of the nanocomposite and create undesired chemical reactivity.

Crystallinity

The degree of structure and order in the ZnO nanoparticles' atomic arrangement is referred to as crystallinity. Better qualities including increased photocatalytic activity, greater mechanical strength, and stronger conductivity are typically linked to higher crystallinity levels.

3.4. Orientation of ZnO Nanoparticles on Graphene

The general characteristics of the nanocomposite are mostly determined by the orientation of the ZnO nanoparticles on the graphene surface. Variations in orientations can result in distinct electronic interactions between graphene and ZnO, which can eventually impact several capabilities like as conductivity and photocatalytic activity.

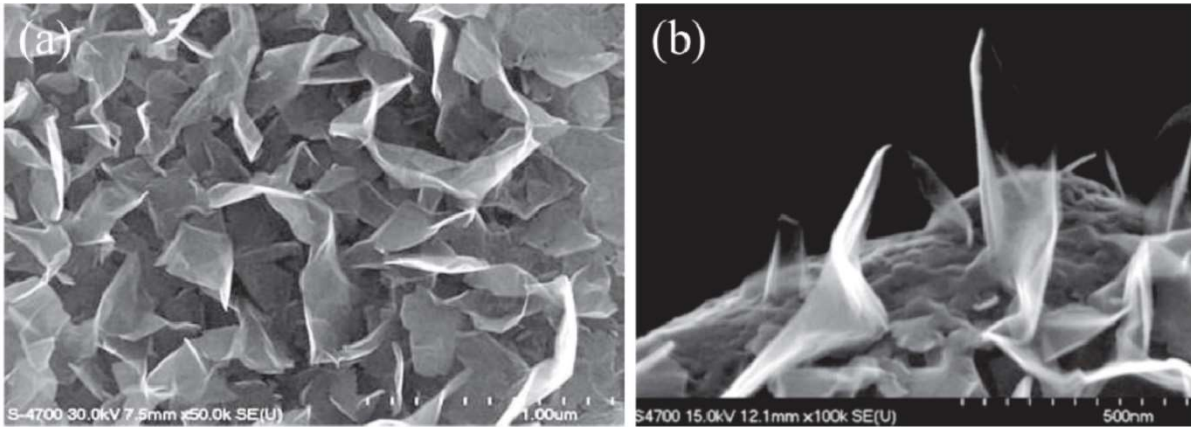


Fig. -4 Orientation of ZnO Nanoparticles on Graphene

Methods for Orientation Assessment:

- X-ray diffraction: The preferred orientation of the ZnO nanoparticles on the graphene surface can be ascertained by analysing the intensity and location of the XRD peaks.
- Electron backscatter diffraction (EBSD): This technique is capable of directly mapping each nanoparticle's crystallographic orientation on the graphene surface.
- Transmission electron microscopy (TEM): The orientation of the nanoparticles with respect to the graphene sheets can be seen in high-resolution TEM pictures.

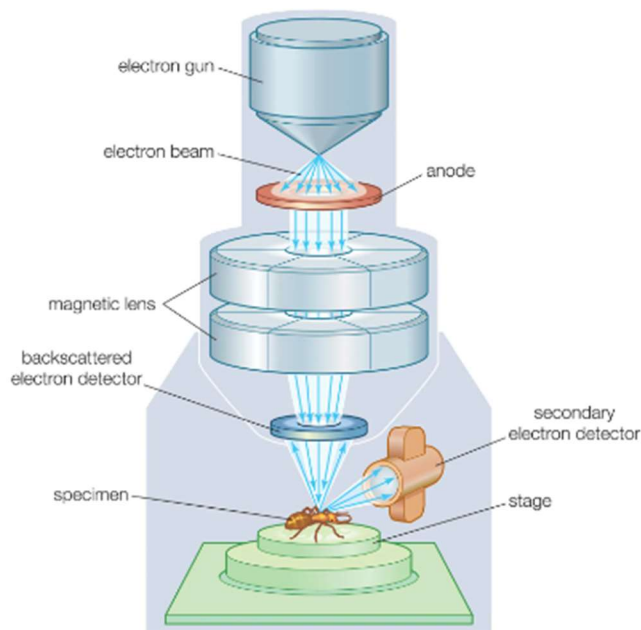
Researchers can modify the characteristics of ZnO nanoparticles on graphene to suit certain needs by comprehending and managing their phase purity, crystallinity, and orientation. To maximise the performance of these promising materials in a variety of applications, such as energy storage, environmental remediation, and optoelectronics, this information is essential.

SCANNING ELECTRON MICROSCOPY (SEM)

4.1. Techniques:

Topographic Imaging: SEM creates high-resolution topographic images by scanning the sample's surface with a concentrated electron beam. This method provides remarkably detailed surface characteristics and imperfections.

Backscattered Electron Imaging: SEM provides contrast in compositional changes, detects backscattered electrons to differentiate materials according to their atomic number, and makes it easier to separate ZnO nanoparticles from graphene.



4.2. Surface Morphology and Topography:

ZnO–Graphene nanocomposites' surface morphology may be seen at micro-to nanoscale levels thanks to SEM. The generated pictures show the material's three-dimensional structure as well as how ZnO nanoparticles are arranged on the graphene sheets. Understanding the nanocomposite's overall texture, grain boundaries, and surface imperfections is made possible by high-resolution topographic pictures, which provide vital information.

4.3. Size and Shape Distribution:

ZnO nanoparticle size and shape distribution inside the nanocomposite can be evaluated using SEM analysis. Researchers can determine the homogeneity and uniformity of the composite material by measuring the diameters of the particles and analysing their forms. To ensure that the appropriate features are realised for particular applications, quantitative data on form and size distribution are crucial for improving synthesis techniques.

4.4. Interfacial Interaction on Graphene Sheets:

SEM is a useful tool for studying the interfacial interaction between graphene sheets and ZnO nanoparticles. Researchers can see the degree of contact, adhesion, and dispersion of nanoparticles on the graphene surface by closely scrutinising the high magnification photographs. Variations in the contrast and morphology at the interface provide indicators of the quality of the interaction,

which is important for adjusting the ZnO–Graphene nanocomposite's mechanical and electrical characteristics.

When examining the interfacial interaction between ZnO nanoparticles and graphene sheets, SEM offers important insights. Through high magnification examination of the photos, scientists can see how much contact, adhesion, and dispersion there are between the nanoparticles on the graphene surface. In order to customise the mechanical and electrical properties of the ZnO–Graphene nanocomposite, variations in contrast and shape at the interface provide visual indications about the quality of contact.

TRANSMISSION ELECTRON MICROSCOPY (TEM)

5.1. Principles: ZnO–Graphene nanocomposites can be effectively analysed using transmission electron microscopy (TEM), a powerful technology that operates on the principle of electrons travelling through a thin material. The transmitted electrons that result from the interaction between the electron beam and the sample form an image that allows for detailed nanoscale investigation. TEM is a highly valuable tool for examining the interior structures and interfaces of nanomaterials due to its great resolving power.

5.2. High-Resolution Imaging: One of the things that sets TEM apart is its ability to obtain high-resolution images at the atomic level. By using a concentrated electron beam with wavelengths significantly shorter than visible light, TEM overcomes the diffraction limit and allows for the observation of individual nanoparticles, crystal lattices, and other minute structural details in ZnO–Graphene nanocomposites. This degree of accuracy is necessary to comprehend the many facets that go into the composite's overall characteristics.

5.3. Internal Structure Analysis:

Comprehensive internal structural study of ZnO–Graphene nanocomposites is made easier with the use of TEM. It reveals the size, shape, and distribution of ZnO nanoparticles as well as their order and organisation inside the graphene matrix. Furthermore, TEM helps researchers identify interfaces, flaws, and any amorphous regions that may be present, which further advances our understanding of the structural dynamics of the composite.

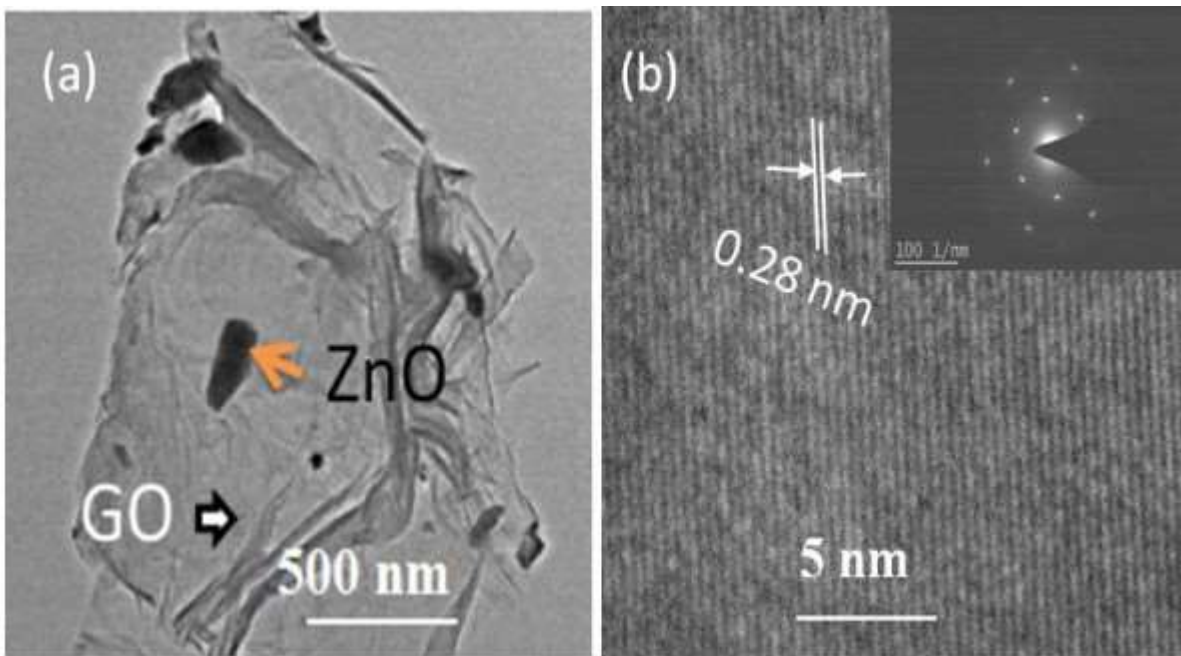
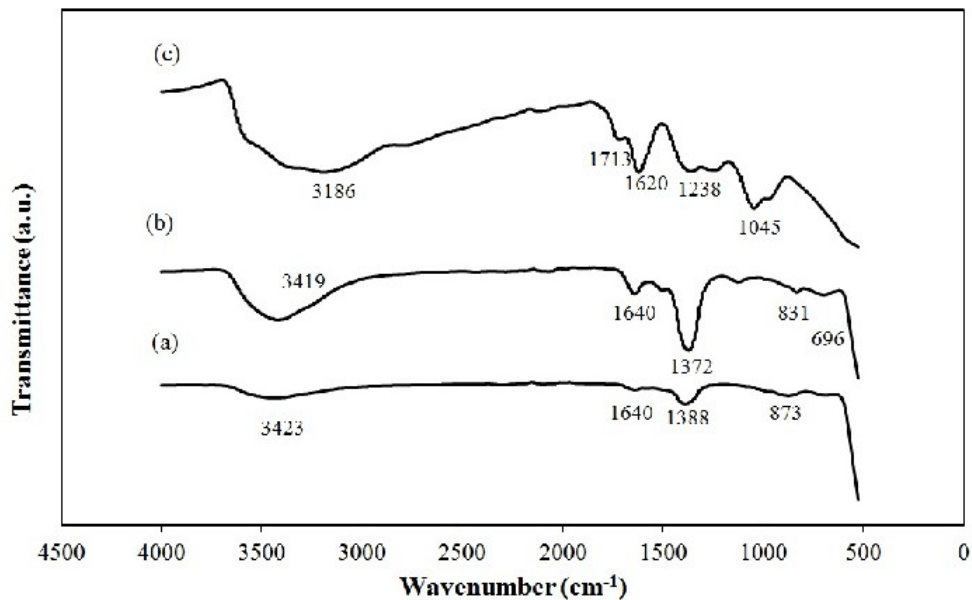


Fig. - 5 TEM image of graphene-ZnO nanocomposites

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Principles: FTIR examines how matter and infrared rays interact. It gathers data on the molecular makeup and functional groups by measuring the absorption, transmission, and reflection of infrared light.



Graph -1 FTIR spectra of (a) ZnO, (b) ZnO/rGO and (c) GO.

ZnO-Graphene nanocomposites have functional groups that are excellently identified by FTIR, which provides a comprehensive molecular signature of the sample. Various functional groups produce characteristic peaks on the FTIR spectrum by absorbing infrared light at different frequencies. These peaks can be analysed to identify the different kinds of chemical bonds that are present and to identify the functional groups that make up the composite.

When it comes to ZnO–Graphene nanocomposites, FTIR makes it possible to identify the functional groups connected to both graphene and ZnO. For instance, functional groups on graphene that include oxygen, such as hydroxyls or carboxyl's, may show particular peaks, and ZnO's FTIR spectrum may show distinctive peaks linked to metal oxides. These peaks' overlap in the composite spectrum shows that functional groups from both components are present, which is important information for customising the material to specific applications.

APPLICATIONS

ZnO-Graphene nanocomposites possess remarkable photocatalytic capabilities, making them ideal for air and water purification. When exposed to light, the nanocomposite breaks down contaminants, offering a viable solution for environmental cleaning. Additionally, the nanocomposite's large surface area and improved charge separation enhance photocatalytic efficiency, further addressing environmental challenges.

ZnO-Graphene nanocomposites find promising applications in the textile industry due to their unique properties. Their antimicrobial and anti-fouling properties enable the development of textiles resistant to bacterial growth and stains, enhancing hygiene and extending product lifespan. Furthermore, the nanocomposite's photocatalytic activity offers self-cleaning functionalities, reducing the need for harsh chemicals and laundering processes.

The combination of photocatalytic and antimicrobial properties in ZnO-Graphene nanocomposites presents a significant advantage for the textile industry. By incorporating these nanocomposites into fabrics, manufacturers can achieve self-cleaning and antimicrobial functionalities, promoting hygiene, reducing environmental impact, and enhancing the overall quality and durability of textiles.

Research efforts are ongoing to further explore the potential of ZnO-Graphene nanocomposites in the textile industry. By tailoring the nanocomposite's structure and composition, researchers aim to optimize its performance for specific applications, such as odor control, wrinkle resistance, and UV protection. The versatility and promising properties of ZnO-Graphene nanocomposites suggest a significant impact on the future of sustainable and functional textiles.

Conclusion

The union of zinc oxide (ZnO) and graphene has birthed a captivating landscape of innovation: ZnO-Graphene nanocomposites. This bonding of two remarkable materials has opened a door to diverse applications and unimaginable technological possibilities.

Synthesis techniques, including hydrothermal and sol-gel methods, have paved the way for creating these nanocomposites with tailored properties. Rigorous characterization using sophisticated tools like FTIR and SEM has unraveled their intricate structures, laying the groundwork for precise engineering.

Their remarkable photocatalytic capabilities make ZnO-Graphene nanocomposites ideal for environmental applications, tackling air and water pollution head-on. Their exceptional sensitivity and selectivity translate into high-performance sensors, while their contribution to energy storage advancements allows for better supercapacitors and batteries. The potential extends even further, with applications in photovoltaics for enhanced solar cell efficiency and in biomedicine as promising diagnostic tools and drug delivery systems.

Looking towards the future, optimization of synthesis techniques remains a key focus, aiming for greater control over the nanocomposites' properties. Green synthesis methods, emphasizing environmental responsibility, are gaining traction, aligning with the global imperative for sustainability.

ZnO-Graphene nanocomposites hold immense potential for addressing critical global challenges. They can play a pivotal role in developing efficient solar cells and energy storage devices, paving the way for a sustainable energy future. Moreover, their biocompatibility opens doors for advancements in diagnostics and drug delivery, improving healthcare outcomes.

By bridging the gap between scientific exploration and technological development, ZnO-Graphene nanocomposites offer a glimpse into a future brimming with innovation and progress. As we continue to unravel their secrets and unlock their full potential, we pave the way for a more sustainable and technologically advanced world.

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