GREEN ROUTE SYNTHESIS AND CHRACTERIZATION OF ZINC OXIDE NANORODS AND ITS COMPOSITE WITH PVA

A DISSERTATION

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF THE DEGREE OF

MASTER

IN PHYSICS

Submitted by:

PARISHA

(2K20/MSCPHY/38)

Under the supervision of:

PROF. NITIN. K. PURI

DEPARTMENT OF APPLIED PHYSICS

DELHI TECHNOLOGICAL UNIVERSITY

(Formerly Delhi College of Engineering)

Bawana Road, Delhi-110042

MAY,2022

DECLARATION

I hereby certify that the work, which is presented in the report entitled "Green Route Synthesis and Characterization of Zinc Oxide Nanorods and its Composite with PVA" in fulfilment of the requirement for the award of the Degree of Master of Science in Physics and submitted to the Department of Applied Physics, Delhi Technological University, Delhi. It's an authentic record of our own, carried out under the supervision of PROF. Nitin K. Puri. The work presented in this report has not been submitted and is not considered for the award for any other course/degree of this or any other Institute/University.

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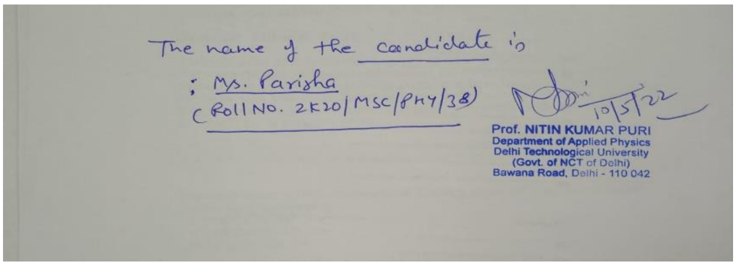

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Parisha
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List of Symbols and abbrieviations

1. ZnO: Zinc Oxide
2. XRD: X-Ray Diffraction
3. FTIR: Fourier Transform Infrared Spectroscopy
4. SEM: Scanning Electron Microscopy
5. NPs: Nanoparticles
6. λ: Wavelength of X-Ray
7. β: Full width Half maxima

Abstract

In this work, I have successfully synthesized Zinc Oxide Nanorods (ZnO-NR) via the Green Synthesis route. Green route has been incorporated to synthesize ZnO-NR and reduce the usage of toxic precursors as well as toxic by-products. Synthesis is carried out using Zinc Nitrate (Zn $(NO_3)_2.6H_2O$) as a precursor and aqueous extract of Osmunda Regilis leaves. The plant extract of Osmunda Regilis leaves has a significant role as a reducing agent because of the presence of polyphenolic compounds. The synthesized ZnO-NR are then characterized by various characterization techniques- Scanning electron microscopy (SEM) X-Ray Diffraction (XRD), , Fourier transform infrared spectroscopy (FTIR), U-V Visible Spectroscopy (UV-Vis). XRD results show that the as-synthesized ZnO sample is extremely crystalline, having a wurtzite structure. UV-Vis absorption spectrum represents a typical spectrum for ZnO-NR which gives absorption peak at 375 nm and exhibits band gap of 3.3 eV. Fourier Transform Infrared Spectroscopy (FTIR) shows the victorious chemical bond formation of ZnO nanorods. SEM micrographs display that formed Nanorods are Crystalline and have a lateral size of nanorods lying between 30-100 nm. Since it is challenging to form a film of bare powder for studying electrical properties therefore we formed ZnO/PVA composite thin film. The electrical properties of ZnO/PVA nanocomposite thin film are studied which exhibits the ohmic behaviour.

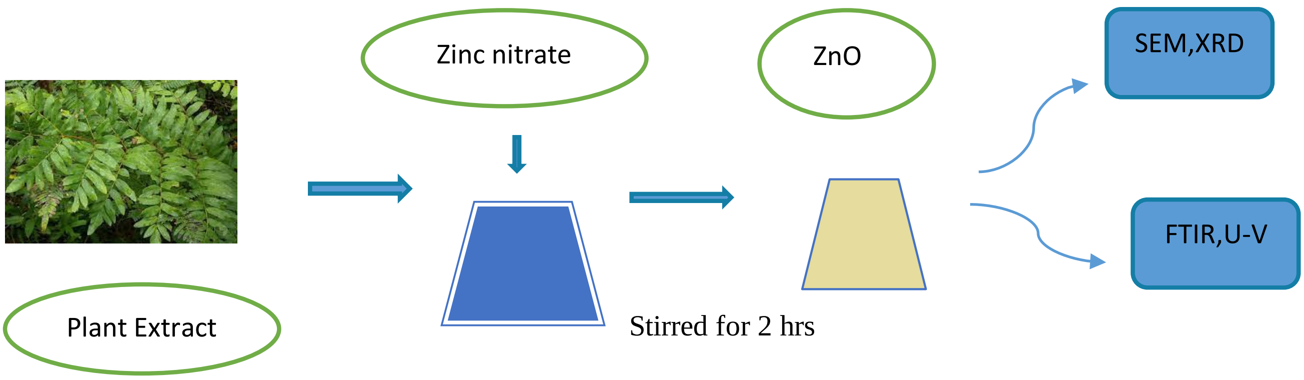

Figure 1: It depicts the whole study of ZnO NPs in this report.

CHAPTER 1

Introduction

Now-a-days nanotechnology is a basic need in every research field. It has been grow day by day. It is considered as one of the major subjects in the branch of applied physics and applied chemistry. It deals with the matter at different dimensions of range between 1-100 nanometers. Nano materials have vast applications due to their size and shape such as nanorods, nanowires etc and structure. Now a days the great pivot on nanomaterials which have major electrical & optical properties. Nanomaterial optical properties, such as absorption, transmission, reflection, and light emission, are dynamic and can differ dramatically from bulk material properties.

By simply altering its shape, size, and surface functioning, a wide range of optical effects can be created for a variety of purposes. Depending on the composition, size, and orientation, this manipulation can be accomplished in a variety of ways. Spherical gold nanomaterials with a diameter of 25 nm seem green, whereas those with a diameter of 100 nm appear orange. In a similar vein, spherical gold nanomaterial with a diameter of 100 nm appears orange, while spherical silver nanomaterial with a diameter of 100 nm seems yellow.

The colour seen in nanomaterials is because of the surface plasmon resonance phenomenon, this generally happens when the nanomaterial's outer electron band resonates along with light wavelengths. Nanomaterials' optical properties are one of the most important, and they can be find using a lot of spectroscopic techniques. The energies of the largest molecular orbital that is occupied, which is a valence band, and the lowest molecular orbital which is unoccupied, which is effectively the conduction band, are most influenced by reduced dimensionality in the electronic structure of small nanoclusters.

The conductivity of a bulk or massive substance is unaffected by parameters such as diameter, cross section area, and change in the conducting wire. However, it has been discovered that the conductivity of carbon nanotubes varies as the cross section area changes. When a shear strain (in plain terms, a twist) is applied to a nanotube, the conductivity changes as well.

Among several nanoparticles, ZnO is considered as one of the versatile semiconductor which act as transparent conductive oxide which opens a lot of applications for it like if we do coating on solar cell to rise its efficiency. Also it has large band gap about (3.2-3.8eV) and crystalline size of (8-30 nm) and It display optical and Luminescent properties in UV – Vis.

It might come as a revelation, but the rubber places utilizes major part of the Zinc Oxide created. In repairing of rubber, ZnO is stirred along with stearic acid to make shoe soles, and hockey pucks also It is utilized as a layer of buffer in Copper Indium Gallium Selenide solar cells, which is a most needy application. The consequence of thickness of ZnO production of power ability for the cells is the pivot of distinct recent inspection.

In ZnO antibacterial and deodorising properties are also observed. As an outcome, it is also used in medical products such as powder of babies & lotions to tend things like diaper rashes, skin problems. It is also used in sunblocks that can be clearly seen on the nose as well as on the lifeguards lips at the beach because of its reflecting characteristics.

High-quality Zinc Oxide has an rousing future ahead of it. Non-medicinal applications could transcend recent medicinal ones. Piezoelectricity and Sensors of Zinc Oxide Nanorod are promising directions to watch in the near future..

Zinc oxide is a frequent ingredient in medicinal ointments for treating skin irritations. Zinc oxide has recently found applications in semiconductors, concrete, ceramic and glass compositions, and even cigarette filters. ZnO is a member of II-VI semiconductor group that has a broad bandgap. The semiconductor's neighbour doping is n-type, that is caused by oxygen vacancies. Excellent transparency, large electron mobility, a wide band gap, and proper room-temperature luminescence are also benefits. Because of these qualities, ZnO is being used in a range of new applications, including opaque electrodes in liquid crystal displays as well as energy-saving and electronics such as thin-film transistors and LED's i.e. light-emitting diodes.

Various methods are used for the Synthesis of ZnO Nps like Sol-Gel Route, Hydrothermal, Precipitation method, chemical vapor deposition, Green Synthesis method. These types of approaches requires high energy and High temperature and it involves poisonous chemicals which affects biological methods badly. Among them Green approach is considered as one of the advantageous approach due to eco –friendly approach also they are clean and safe and "cost-effective". Also, biological approach includes natural things which act as capping agent and stabilizing agent.

Here, we describe the green synthesis of ZnO nanorods using plant extract of Osmunda Regilis and Zinc salt (Zinc Nitrate) as a precursor. Osmunda regilis commonly known as Royal Fern is a species of deciduous fern with medical properties local to Africa, Europe, and Asia which is growing on the seaboard. It belongs to the family Osmundaceae. Its configurations are rounded in shape, moisture-loving, and natural. It accommodates various phytochemicals which show the presence of flavonoids, phenols, and triterpenoids and acts as a reducing agent. They have metabolites and some properties which have the capacity that can convert metal ions into nanoparticles. Several substances like sugars, alkaloids, phenolic acids, and polyphenols can reduce metal ions' reduction value or strength. It is a single-step procedure and that is why it needs less amount of energy. It can control the growth of the crystal and the size of the particle because they act as a capping agent. It is done under the presence of ambient temperature, pressure, pH value which reduces the toxicity of chemicals [2]. To demonstrate the electrical properties of ZnO, a nanocomposite of ZnO/ PVA has been made.

CHAPTER 2

Advantages of green synthesis method

Here Plants, fungi, bacteria and algae are used for the synthesis purpose because they decrease the value of metal ions in nanoparticles.

- They have metabolites and some properties which have capacity that is able to convert metal ions into nanoparticles.
- Several substances like sugars, alkaloids, phenolic acids and polyphenols that can reduces metal ions reduction value or strength.
- It is single step procedure and that is why it needs less amount of energy.
- It can control the growth of crystal and size of the particle bcz they act as capping agent.
- It is done under near ambient temperature, pressure and pH value.
- It reduces the toxicity of chemicals.
- It raise the efficiency of the process.
- It provides a precise, simple single step and eco -friendly way which reduces the bad/side effects of chemical and physical methods.
- There is no need of capping and stabilizing agent particularly because the plant itself acts as both the agents i.e. capping and stabilizing agents.
- It needs low protection because if made once then we can store it for future use at cold place and it is cost- effective and therefore it is stable method.
- Besides this, biological applications can be approached by this method such as antibacterial activity of the material.
- It requires low cost and nontoxic materials, and is operated under ambient conditions and accessibility of renewable raw materials.
- Hence, It is instantaneous approach for pollution free environment.

Preparation of zinc oxide NPs

Chemical reaction:

- $Zn(NO_3)_2 + 2NaOH \rightarrow Zn(OH)_2 + 2NaNO_3$

Zinc nitrate | Sodium hydroxide | Zinc Hydroxide | Sodium nitrate

- $Zn(OH)_2 \xrightarrow{\Delta} ZnO + H_2O$

Zinc hydroxide | Zinc Oxide | Water

- In this reaction when zinc nitrate is added with sodium hydroxide then it gives zinc hydroxide and sodium nitrate.
- And after that when zinc hydroxide so produced from the last reaction get heated which gives zinc oxide and water.

CHAPTER 3

EXPERIMENTAL

Preparation of plant Extract:

Fresh leaves which was extracted from Delhi technological university campus, that is Osmunda Regilis which is phenolic rich and have some properties like antimicrobial, antibacterial, medicinal due to which I used this plant in synthesis of zinc oxide nanoparticles. Firstly this leaves were collected and then wash this with tap water twice or thrice then after dried this leaves at room temperature for two or three days and then grind this to make it in powder form with the help of mortar pestel after that 1.5g of leaves powder was added into 37.5g of deionized water further the solution was boiled at 60^0C for 15 minutes before filtering through filter paper and then it was stored at cold place for future use.

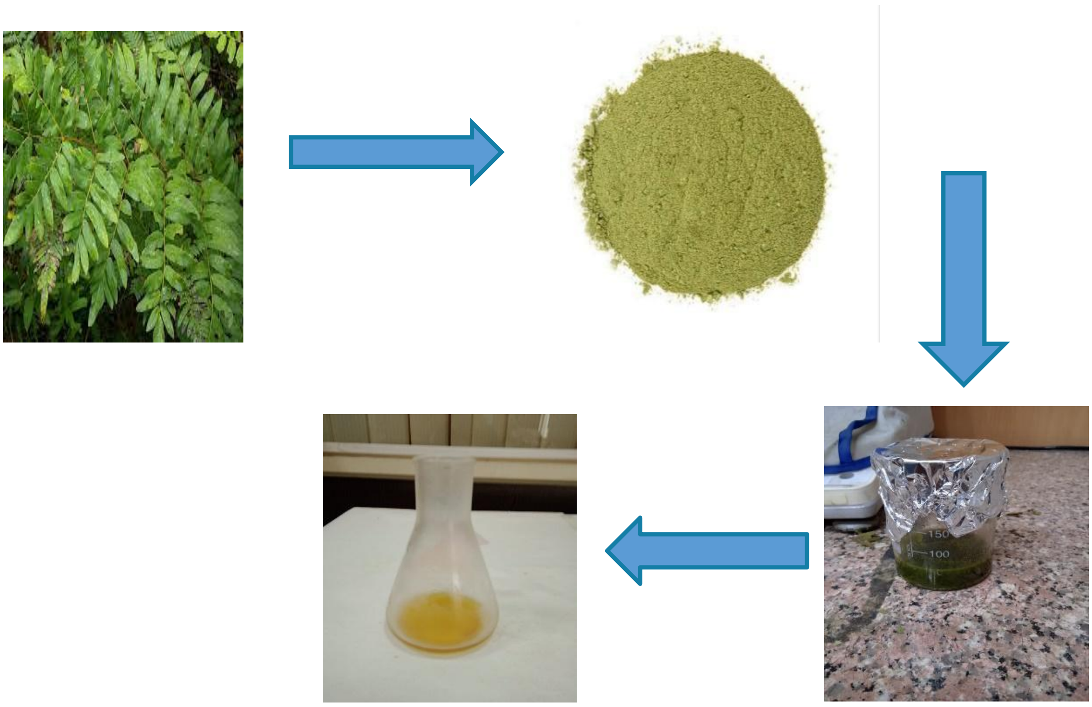

Figure 2: It depicts the aqueous plant extract from Osmunda regilis leaves.

Methodology:

In Biological/Green synthesis of Zinc nanoparticles with help of zinc nitrate as a precursor, 25 mL of 0.05M $Zn(NO_3)2.6H_2O$ solution was assorted with 4 mL of Osmunda Regilis (Royal Fern) leaves aqueous extract solution. Then stirring was done for few hours (around 2 hrs) using magnetic bead. Then after that 0.02M NaOH was added dropwise to stick out pH 12. The mixture was again stirred for 1 hour extra at 250 rpm till we get a product that is solid in form is obtained with a light yellow in color. The precipitate was purified 3-4 times with the help of deionized water and ethanol and then centrifuged at 6000 rpm for 10 min in two go. Then finally white color product was obtained which was further dried for 2 hrs in an oven at 80°C.

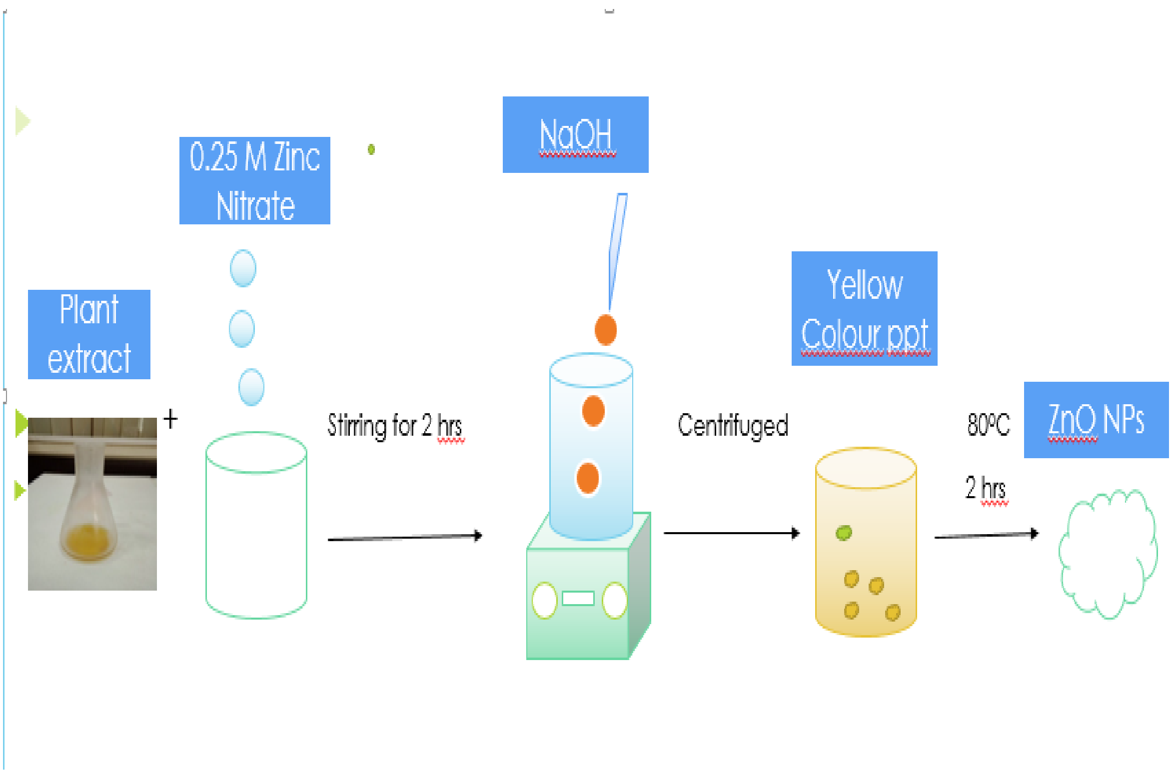

Figure 3: This figure depicts the synthesis process of ZnO Nanorods

CHAPTER 4

Results and discussions

XRD:

XRD is a method for deciding a material's primary structure of a crystal; this permits for the confirmation of a structure and crystallinity of a sample, its structure that gives no such chemical data. The lattice parameters of material, the given crystal's orientation , the stress which is found in crystalline regions. It's a bulk characterization tends that provides mean diffraction pattern for the area which has been measured. It is a non-destructive method that can be executed at proper temperatures and pressures.

X-ray source of Cu K_α radiation i.e 1.54 is used for the XRD examination. It uses the Bragg method to determine and verify unknown crystalline stuff. Based on the requirements of specimen's, several values like scan step size, its collecting time, range of material, tube voltage of X-ray, and current should be set properly. For phase verification of a few variety of crystalline phases in concrete illustration, the standard database i.e. JCPDS database for the XRD pattern is verified.

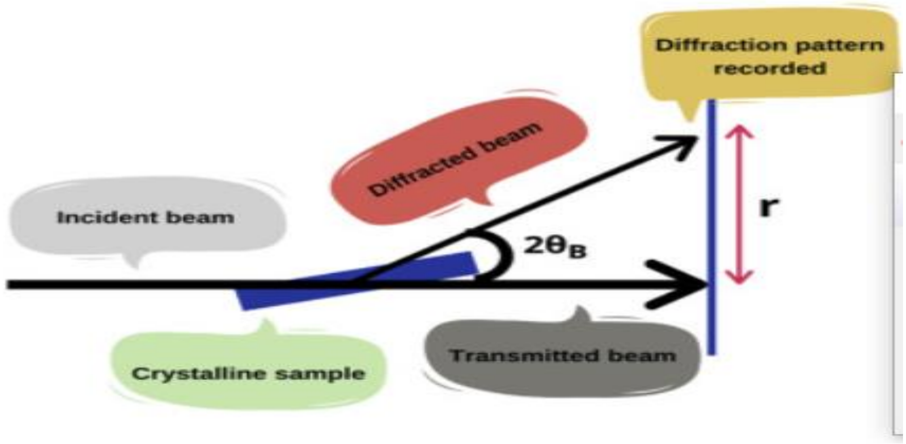

Figure 4: Illustration of X-Ray Diffraction

XRD result:

XRD pattern of synthesized ZnO nanorods which represents the composition of the nanoparticles that we synthesized is crystalline. The keen diffraction peaks remarked at 2θ values which are confirmed from **(JCPDS card no-00-001-1136)** 31.88^0, 34.52^0, 36.36^0, 47.56^0, 56.64^0, 62.96^0, and 66.6^0. The peaks are listed as (100), (002), (101), (102), (110), (103) and (112) diffraction lattice planes which verify the hexagonal wurtzite structure for the ZnO nanorods we synthesized. The crystalline size i.e. t is found using Debye Scherer's formula-

$$D = \frac{0.9\lambda}{\beta Cos\theta}$$

Where λ= wavelength of X-ray i.e. 1.54 Å, β= Full-width half maxima (radian), θ is Bragg's angle [2dSinθ =n λ].

Sample	d-spacing (nm)	FWHM (radian)	Estimated Crystalline Size (nm)
1.	11.52	0.227	37.4

► Table 1: Shows details of XRD analysis of synthesized Zinc Oxide Nanorods

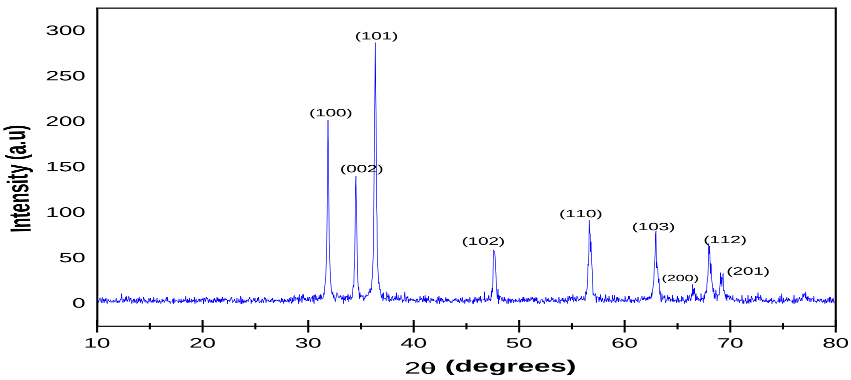

Figure 5: Graphical depiction of XRD Spectra of prepared ZnO Nanorods

UV-Vis spectra :

UV spectroscopy, also called as UV–visible spectrophotometry (UV–Vis or UV/Vis) is the whole study of absorption as well as reflectance spectroscopy in the U-V and its nearby visible parts of the E-M spectrum that makes the utility of visible and neighboring light. The color of the materials jumbled is directly overblown because of their absorption in the visual spectrum. Molecules and atoms that undergo under transitions that is electronic in this region of the spectrum. Absorption spectroscopy is identical to fluorescence spectroscopy from that it can find transitions takes place from ground state to the excited state, but fluorescence finds transitions takes place from the excited state to the ground state.

The wavelengths of absorption peaks are useful in findingg the functional groups present in a molecule since they can also be associated with the types of bonds in a given molecule. For conjugated materials, the Woodward–Fieser criteria are a collection of empirical data which is used to find max, the wavelength of the most enormous UV/Vis absorption.

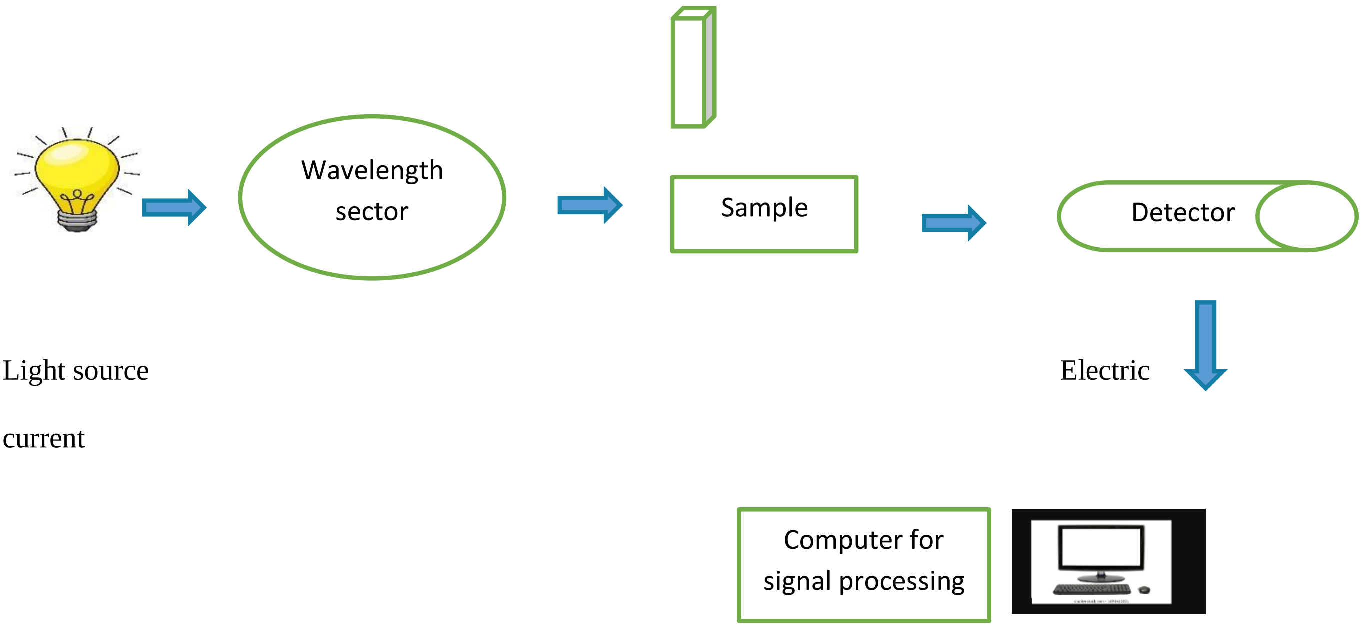

Figure 6: UV- Vis spectroscopy functionality

UV- Visible Spectroscopy result:

UV–visible spectra of synthesized zinc oxide nanorods are shown in the above figure. The peak is at around 375nm because of high binding energy at normal/room temperature. Also, we know that by absorption spectroscopy the bandgap increases when the size of the particle decreases as ($t \propto 1/E_g$) where E_g is the bandgap gap of ZnO NPs calculated from the above formula-

$$(\alpha E)^{1/n} = \beta (E-E_g)$$

Where the transition mode power factor is n, E_g is the optical bandgap energy, the band tailing parameter is β, and E is calculated from the above formula i.e. E = hν, where h is the Planck's constant, ν is frequency of UV – Vis spectra. ZnO is a direct bandgap material for allowed transitions therefore n=1/2.

From the above formula, the bandgap is calculated to be 3.2 eV. Whereas, the actual band gap for ZnO nanorods is found to be 3.8 eV which is larger than the bandgap we found this is how we see that the particle size is inversely related to the bandgap because if the particle size is less than it enlarges the energy bandgap.

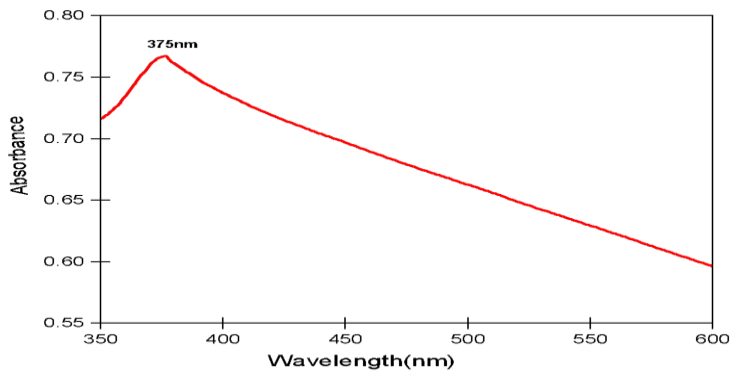

Figure 7: Graph representing U-V Visible spectra of ZnO nanorods

Fourier transform Infrared Spectroscopy:

FTIR (Fourier-transform infrared spectroscopy) is a technique used for determining an infrared spectrum of a solid, liquid and gas's absorption. An FTIR spectrometer get large-resolution spectral data compares to a high spectral number at same time. This gives it a big hump compares to a dispersive spectrometer, which only finds intensity over a small range of wavelengths at a given time. The use of Fourier transform spectroscopy to derive the same information is less apparent. Instead of using a single sample wavelength's light, this method has a beam that have multiple frequencies of light at similar time and analyses how much beam is taken by the sample. This technique is taken many times in a short period. After that, a system uses all of this information to deduce absorption at each different wavelength by working backward.

DEVELOPMENTAL BACKGROUND

The Perkin-Elmer Infracord, introduced in 1957, was the first spectrophotometer that have a low cost which is efficient of laying down an infrared spectrum. The wavelength range covered by this device was 2.5 m to 15 m . The lower wavelength limit was set so as to surround the greatest familier molecular vibration frequency. prism was the dispersion element and is manufactured from a single crystal of rock-salt i.e. sodium chloride, which becomes transparent at wavelengths larger than around 15 m, imposing an upper limit; this spectral area was dubbed the rock-salt region. Later devices employed potassium bromide prisms to increase the range to 25 m (400 cm^{-1}) and cesium iodide prisms to increase the range to 50 m (200 cm^{-1}). The far-infrared region, which extends beyond 50 m (200 cm1) and combines with the microwave zone at very long wavelengths, became known as the far-infrared region. Because salt crystals are opaque in this area, Calculations in the far-infrared necessitated the production of precisely governed diffraction gratings to substitute prisms as a dispersing elements. Due of the low energy of the radiation, more sensitive detectors were made necessary. The Golay detector was one of them. Another challenge is the requirement to eliminate atmospheric water vapor, which has a strong pure rotational spectrum in that region. Far-infrared spectrophotometers was inconvenient, slow as well as costly to use. The benefits of the Michelson interferometer fully understooded, further there were significant technological challenges to solve before commercial equipment could be developed.

Figure 8: FTIR appartatus

Fourier Transform Infrared spectroscopy Result:

FT-IR studies of Osmunda Regilis aqueous plant extract and product of synthesized ZnO-NR as shown in Figure 5. Infrared studies were achieved to find out the purity, nanoparticles' nature, and also gives the proximity of phytochemicals that are present in the material. The phytochemicals such as phenols, amines, alcohols, and carboxylic acids that can also interact with the z surface of zinc and support the maintenance of ZnO-NR. The peak at1196 cm^{-1} specifies that it is a C-N stretch which specifies that there is a presence of amines. The peak at 1637 cm^{-1} corresponds to Zn-O stretching and deformation vibration. The peak at 3434 cm^{-1} gives the OH stretching vibrations which indicate there is the presence of the carboxylic acid functional group.

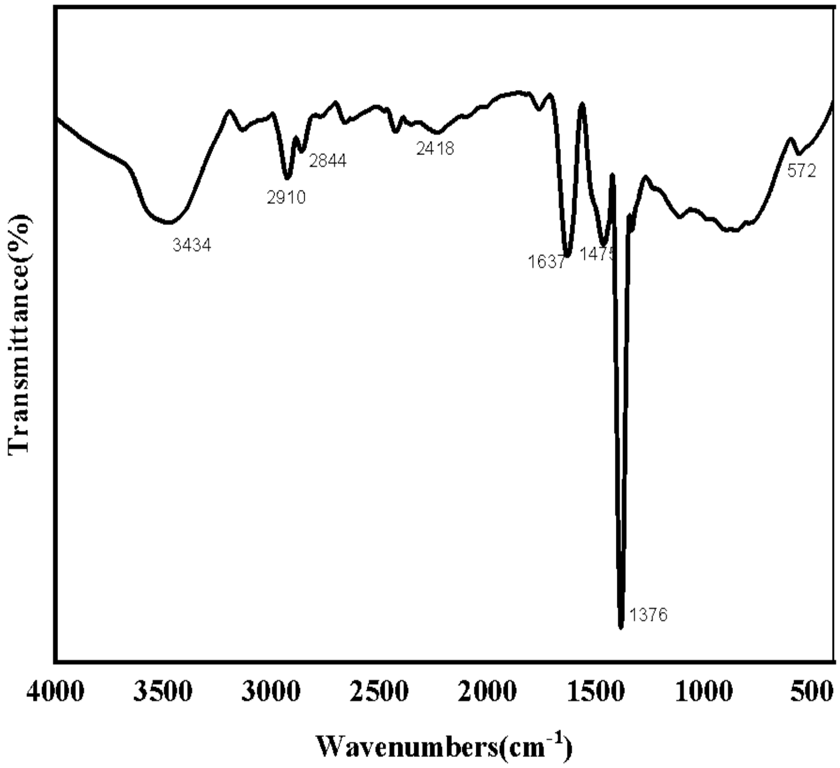

Figure 9: FTIR spectra of ZnO nanorods.

Scanning electron Microscopy:

The electronic console and the electron column are the two major components of the SEM instrument instrument modifications such as filament current accelerating voltage focus magnification brightness and contrast can all be made using the electronic consoles control knobs and switches the fei quanta 200 is a cutting-edge electron microscope that pairs an electronic console with a computer system eliminating the need for a bulky console with control knobs crts and a device that is captured the mouse and keyboard are used to access all the major controls on the computer system instead of the knobs control as well as switches seen on scanning electron microscopes that used previously the operator simply needs to be freindly with the software that operates that instrument the picture drawn by the SEM is typically seen on crts also on the electrical console but along with fei the image is displayed on a computer monitor captured images could be saved in digital format or printed right away.

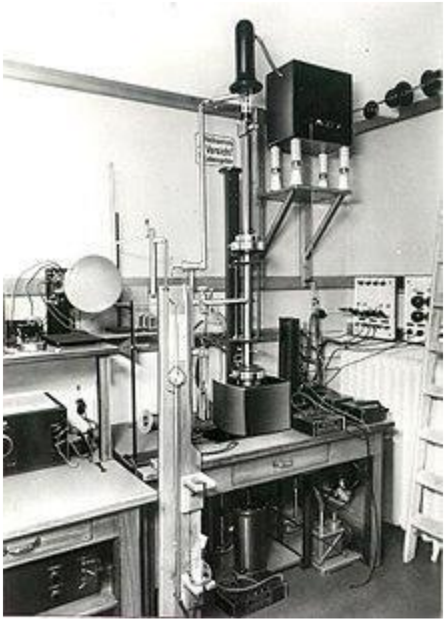

Figure 10: Andrenne's First SEM

The column of electronics must be undergoing vacuum at a pressure of minimum $5x10^{-5}$ Torr for a SEM to create a regulated beam of electrons. For a variety of causes, a high vacuum pressure is needed. To begin, the current which is passing across the filament leads it to achieve temperatures of roughly 2700K. In the presence of air at atmospheric pressure, a hot tungsten filament will oxidise and burn out. Second, the proper operation of the column optics necessitates a somewhat clean, dust-free atmosphere. Third, inside the column, air particles and dust might interfere with and block electrons before they have to reach to the specimen in the chamber of sample. A vacuum system is required to maintain enough vacuum pressure inside the column.

Microscopy was originally predicated for the usage of a light microscope, which could yield resolutions of specimen of 0.2 μ. Larger resolutions can be achieved by using an electron source rather than light acting as the illumination source, that allows for resolutions of roughly 25 Å. Because of the nature of electron beam that interacts with specimen, there are a range of signals that could be used for offer details on properties at and near the specimen surface's when using electrons.

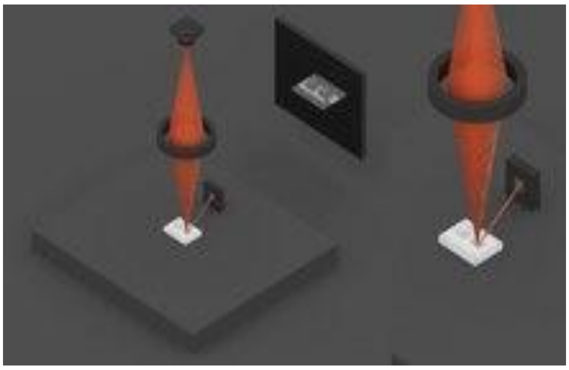

Figure 11: Operating Principle of SEM

Figure 12: SEM with opened Chamber

Scanning Electron Microscopy Result:

Figure 13 shows the SEM images of ZnO Nps, which clearly shows the synthesized ZnO Nanoparticles are nanorods and their size lies between 30-100nm having a crystalline structure, high magnification figure (13a) and low magnification (13b) SEM images are taken. The image with low magnification shows uniform ZnO nanorods with uniform yield. The image with high magnification nanorods surface is plane. Very minute second particle growth components are interposed.

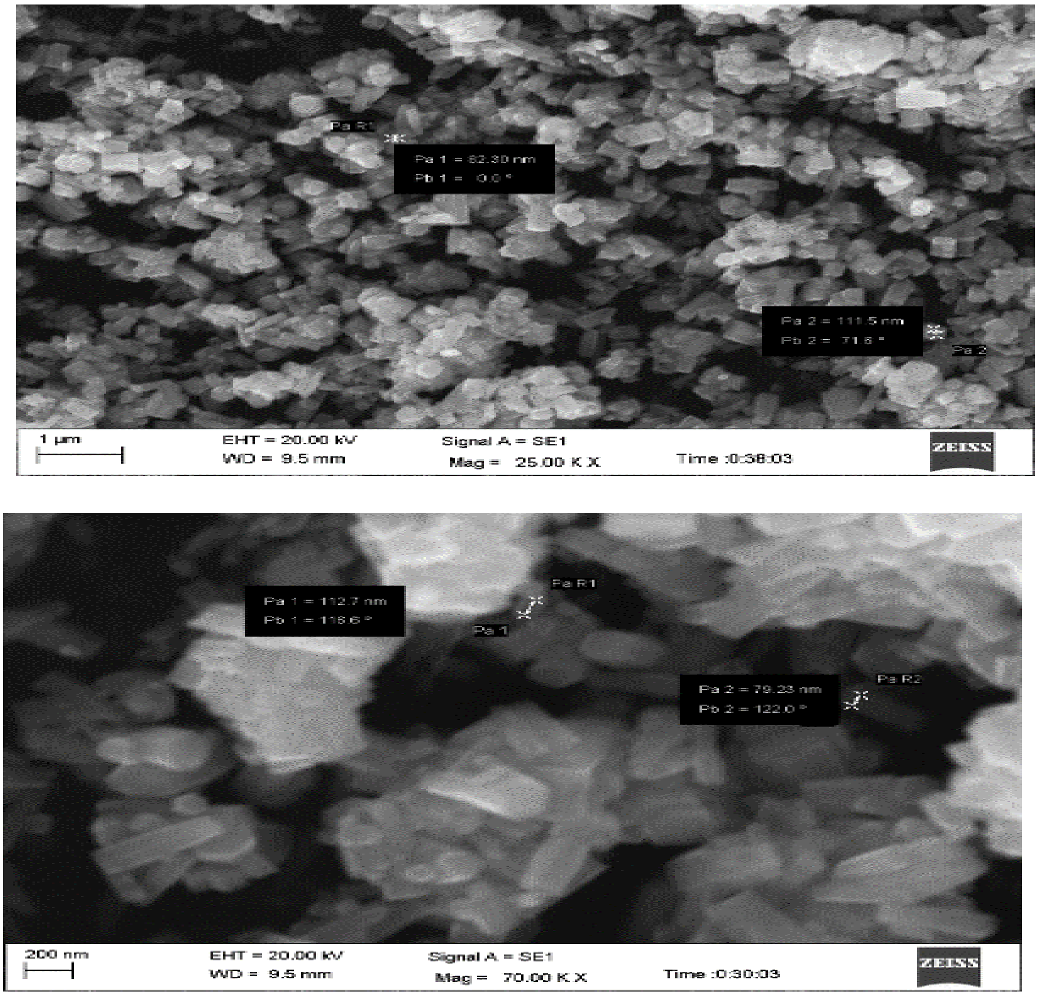

Figure 13: SEM images of ZnO Nanorods (ZnO-NR) (13a) SEM images showing the particles at a scale of 1 µm and (13b) SEM images showing the particles at a scale of 200 nm.

CHAPTER 5

Electrical Properties

Zinc oxide is one of the important semiconductors in group II–VI. It's a 3.37 eV direct energy gap oxide semiconductor with a large bandgap. ZnO is chemically and mechanically stable, as well as harmless and abundant material. Transparent-conducting oxides that is based on ZnO have recently received a lot of attention. ZnO is one of the most assured materials for UV optoelectronic devices, as well as optical and display technologies for the future generation. ZnO is an important material for heat reflectors in gas stoves and thin film electrodes in non crystalline silicon solar cells due to the immediate occurrence of both high optical transmittances in the visible range and low resistance. ZnO is a semiconducting and optical material waveguide that is utilized in sensors, acoustic surface devices, transparent electrodes, and solar cells that belongs to the hexagonal wurtzite class. Controlling the physical properties of ZnO as they change due to various causes including doping and temperature growth is critical for the efficient operation of devices based on ZnO structures. For applications in light-emitting devices, the presence of both (n and p) conduction types is critical.

For their prospective uses in practical domains like field emission displays, solar cells, U-V photodetectors, optical switches, and gas sensing applications, such nanotubes, nanowires, nanorods, and nanofibers have warranted special study. Chemical bath deposition and the sol-gelprocess are two other well-known ways to make ZnO thin films. Spray pyrolysis is one of these technologies that have a wide variety of applications. This process is less expensive, easier, and allows for the production of films with the desired properties for optoelectronic applications. The effect of thickness on structural and optical properties of ZnO films is explored.

Nanostructured materials' unique and fascinating features have sparked a lot of interest among academics who want to learn more about how they might be used in technological applications. Because of their potential uses in the manufacturing of microelectronic and optoelectronic devices, the electronic and optical properties of nanostructured materials have piqued researchers' curiosity. The ZnO films were deposited on silicon, glass, and ITO were accumulated by the drop-casting mesh PVA.

Preparation of thin films :

The ZnO /PVA Nanocomposite films were made by the drop-casting method. Synthesized ZnO was added in weight percent ratio by wt% of 5 to PVA from the formula as given below:

$$a\ (wt\%) = 100\times \frac{wz}{wz+wp}$$

Where w_z and w_p represent the weight of ZnO and PVA.

The Nanocomposite is produced by dispersing ZnO powder into PVA (2g) which is dissolved in 50 ml of DI water and then the solution is kept for stirring for 1 hour at 80^0C to prevent it from any type of agglomeration. Then films were made through the drop-casting method. Lastly, the films were dried in an oven at 80^0C

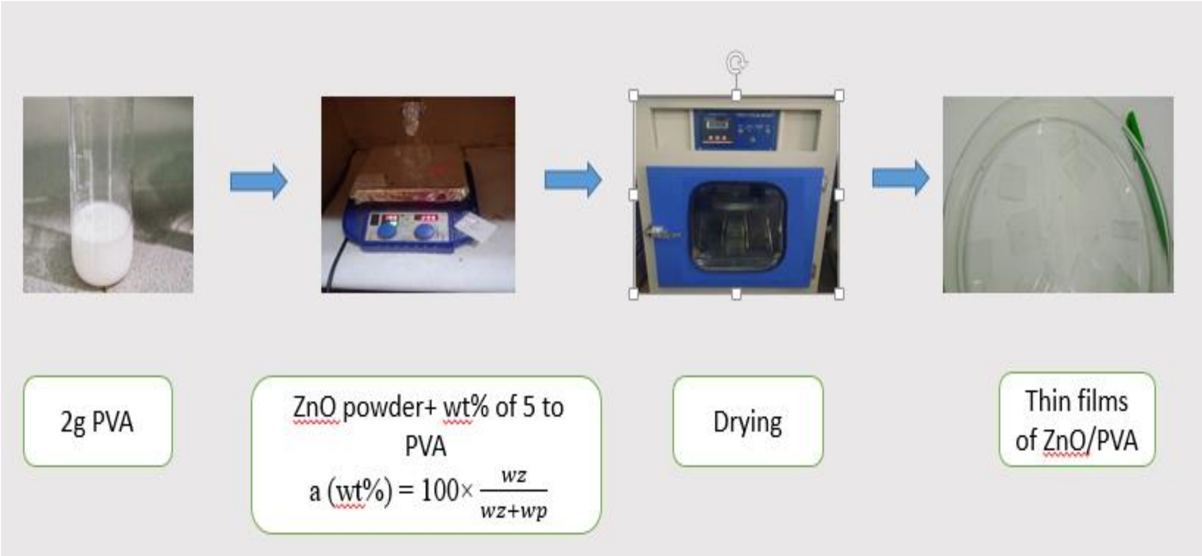

Figure 14: ZnO Nanocomposite with PVA

Why PVA is used:

The unusual and exciting properties of nanostructured materials have piqued the curiosity of academics who want to understand much more about that they may be utilised in technological applications. The electrical of nanostructure materials have aroused researchers' interest due to their prospective applications in the fabrication of microelectronic devices.It's one of the most popular water-dispersed adhesives on the market. Ethylene copolymers include polyvinyl alcohol (PVOH) and polyvinyl chloride (PVC). PVA is utilised as an emulsion adhesive in the production of bags, sacks, and cartons. PVOH is created by hydrolysis of PVA, and the –OH groups impart strong hydrogen bonding, making pure PVOH water soluble. The amount of hydrolysis determines the degree of water solubility.

PVOH is used in unit dosages of detergents, where the complete pack is thrown into the washing machine, and in agrochemicals, when the pack is thrown into a tank of water and combined. The advantages of both of these examples are that a measured dose of substance is utilised, with minimal spillage during decanting and, in the second case, limited worker exposure to a potentially dangerous agent.

The temperature at which composites are mixed, the concentration of glycerine, and the concentration of nanometric zinc oxide all have an impact on product qualities. The formulations were then examined for physicochemical, utility, and microbiological qualities in the final step. According to the results of the testing, the composites show antibacterial properties. This research focuses on the development of bactericidal and fungicidal functional composites based on poly(vinyl alcohol) with the inclusion of zinc oxide nanoparticles. The product is expected to be used on flat surfaces infected by microorganisms, such as floors in locker rooms or baths, or pool overflow grates. The goal is to create a substance that solidifies after application and can be torn off with the dead biological film.

MATERIALS

Poly(vinyl alcohol) (M = 72,000 g/mol, 99.0%), chitosan (M = 100,000–300,000 g/mol, high purity), gelatin (p.p.a.), xanthan gum (p.p.a.), glycerine (d = 1.26 g/cm3, p.p.a.), methyl cellulose (viscosity = 400 cP, p.p.a Sigma Aldrich provided all of the chemicals.

Source meter connections:

From the I-V curve, we obtained of ZnO/PVA nanocomposite thin film were measured at normal temperature as given in Figure, we can easily demonstrate that the graph obeys ohms law from its linearity above 0.5 volts.

I assume you've grown or fabricated thin films on naked glass.

Simply give electrodes to the film (both Glass and film).Nothing more than soldering little wires to a plate. However, soldering is not an option here. As a result, get some high-quality copper thin wire.

On both sides of the wire, remove the insulation.On raw glass, connect one end of the wire with high-grade silver paste.Connect a second wire to the film.Connect terminals to your sample electrodes with Keithley Source Measure Units. It should be measured and analysed.

Figure 15: Source meter connections

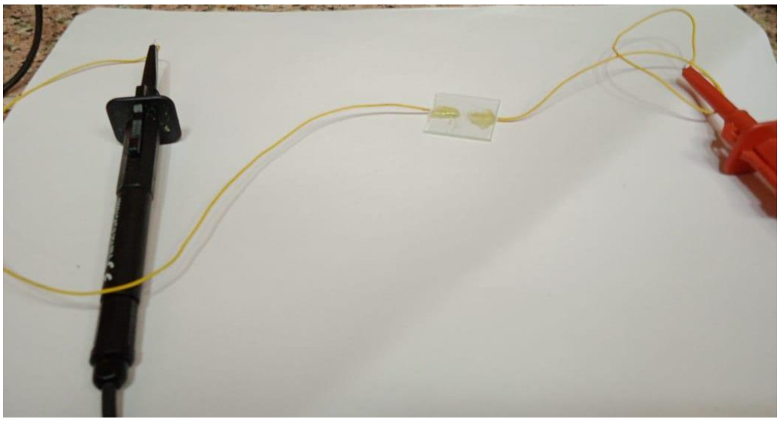

Figure 16: Electrical measurements of ZnO/PVA thin film

Results

From the I-V curve, we obtained of ZnO/PVA nanocomposite thin film were measured at normal temperature as given in Figure, we can easily demonstrate that the graph obeys ohms law from its linearity above 0.5 volts.

As ZnO represents semiconducting behave as PVA i.e Polyvinyl alcohol represents conducting nature which represents its better charge transport through it [3]. This linear nature in the interface of metal and insulators is very salient in the growth of electronic devices with better performance and trustability and it is proved that ZnO is semiconducting in nature and PVA i.e. Polyvinyl Alcohol is Conducting in nature because their arises an intercept which clearly indicates that it obeys ohms law but above 0.5 volts.

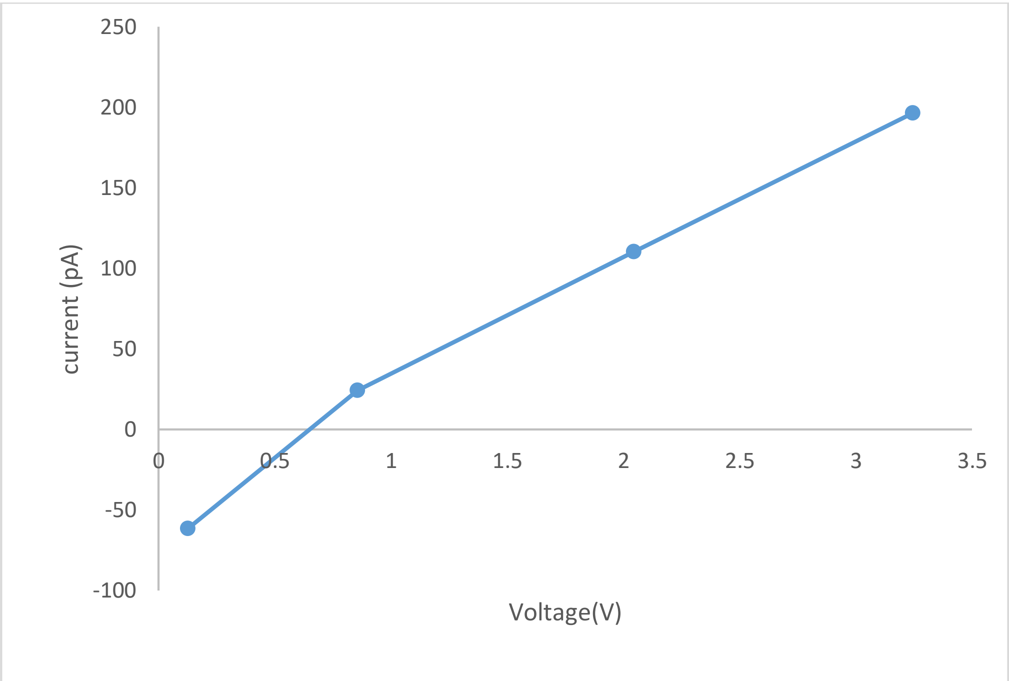

Figure 17: I-V curve of ZnO/PVA nanocomposite thin films

Conclusion

In this work, ZnO-NR was synthesized by Osmunda Regilis leaves aqueous solution and Zinc Nitrate, with NaOH as a precursor. The results of XRD committed the regulation of the synthesis procedure which indicates the creation of crystallite zinc Nanorods along with a hexagonal wurtzite structure. UV–Visible spectrum gives a clear peak at 375 nm, from which we determined the value of band gap i.e 3.2 eV. The average size of synthesized ZnO-NR was 37.4nm. FT-IR studies show the clear evolution of ZnO-NR and contain various phytochemicals which we can determine from peaks at different positions which act as capping and reducing agents for the synthesized ZnO-NR. The I-V characteristics of ZnO/PVA nanocomposite thin film reveal that it is Ohmic nature.

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Green route synthesis and characterization of zinc oxide nanorods and its composite with PVA

Parisha Diwan, Kanika Sharma, Nitin K. Puri *

Nanomaterials Research Laboratory (NRL), Department of Applied Physics, Delhi Technological University, Bawana Road, Delhi 110042, India

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ABSTRACT

In this work, we have successfully synthesized Zinc Oxide Nanorods (ZnO-NR) via the Green Synthesis route. Green route has been incorporated to synthesize ZnO-NR and reduce the usage of toxic precursors as well as toxic by-products. Synthesis is carried out using Zinc Nitrate (Zn $(NO_3)_2 \cdot 6H_2O$) as a precursor and aqueous extract of Osmunda Regilis leaves. The plant extract of Osmunda Regilis leaves has a significant role as a reducing agent because of the presence of polyphenolic compounds. The synthesized ZnO-NR are then characterized by various characterization techniques- X-ray Diffraction (XRD), U-V Visible Spectroscopy (UV–vis), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM). XRD results show that the as-synthesized ZnO sample is extremely crystalline, having a wurtzite structure. UV–vis absorption spectrum represents a typical spectrum for ZnO-NR which gives an absorption peak at 375 nm and exhibits a bandgap of 3.3 eV. Fourier Transform Infrared Spectroscopy (FTIR) shows the victorious chemical bond formation of ZnO nanorods. SEM micrographs display that formed Nanorods are Crystalline and have a lateral size of nanorods lying between 30 and 100 nm. Since it is challenging to form a film of bare powder for studying electrical properties therefore we formed ZnO/PVA composite thin film [8]. The electrical properties of ZnO/PVA nanocomposite thin films are studied which exhibit the ohmic behavior.

Selection and peer-review under responsibility of the scientific committee of the International Conference on Materials, Processing & Characterization.

1. Introduction

Nowadays nanotechnology is a basic need in every research field [1]. The interest of researchers in nanotechnology has been growing day by day. It is one of the important subjects which play a pivotal role in branches of applied physics and applied chemistry. It deals with the matter at different dimensions ranging from 1 to 100 nm. Nanomaterials have vast applications due to their size, shape, and structure. Nowadays the great focus has been shifted towards nanomaterials which show superior electrical, structural & optical properties [2].

Today nanomaterials are in demand because of their reduced size, exceptional surface active sites, and they are progressively being used in various sectors such as biomedical, drug delivery, and photothermal therapy due to their antimicrobial and anticancer properties [3]. Various types of nanomaterials have been extensively used such as metal oxides (ZnO, PdO, PTO) [4], metal nitrides (h-BN, AlN, GaN) [5], Transition Metal Dichalcogenides (TMDs –MoS_2, WS_2, $MoSe_2$, WSe_2). Among them, the great focus is on metal oxides because of their major properties such as optical, electrical, mechanical, and piezoelectric and it is considered the major factor for productive interaction with target molecules.

Among several metal oxides, ZnO is considered one of the versatile semiconductors which act as a transparent conductive oxide that opens a lot of applications [4]. The interesting properties of ZnO when coated on conducting substrate exhibits appreciable efficiency for solar cell and is depicted by the fabrication of quantum-dot sensitized solar cells [6]. Also, it has a large band-gap of about (3.2–3.8 eV) and crystallite size of (8–30 nm). It displays optical and luminescent properties in UV–Visible spectroscopy.

For the synthesis of ZnO nanoparticles, various methods are used like the Sol-gel route, Hydrothermal, Precipitation method, Chemical Vapour Deposition, Green route synthesis method, etc [1,6]. These types of approaches require high energy and high temperature and it involves poisonous chemicals which affect biological methods badly. Among them, the green approach is considered

* Corresponding author.
E-mail address: nitinkumarpuri@dtu.ac.in (N.K. Puri).

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one of the advantageous approaches due to the eco-friendly approach also they are single step, safe, and economical and it has a lot of applications in farming, medical, and industries [7]. Due to the physicochemical nature of plant-based nanoparticles, this method provides an additional advantage of a high lifetime of nanoparticles that prevail over the disadvantages of conventional methods i.e. physical and chemical methods of nanoparticle synthesis. Also, the biological approach includes natural things which act as capping agents and stabilizing agents [8].

Here, we describe the green synthesis of ZnO nanorods using plant extract of Osmunda Regilis and Zinc salt (Zinc Nitrate) as a precursor. Osmunda regilis commonly known as Royal Fern is a species of deciduous fern with medical properties local to Africa, Europe, and Asia which is growing on the seaboard. It belongs to the family Osmundaceae. Its configurations are rounded in shape, moisture-loving, and natural. It accommodates various phytochemicals which show the presence of flavonoids, phenols, and triterpenoids and acts as a reducing agent. They have metabolites and some properties which have the capacity that can convert metal ions into nanoparticles. Several substances like sugars, alkaloids, phenolic acids, and polyphenols can reduce metal ions reduction value or strength. It is a single-step procedure and that is why it needs less amount of energy. It can control the growth of the crystal and the size of the particle because they act as a capping agent. It is done under the presence of ambient temperature, pressure, pH value which reduces the toxicity of chemicals [9]. To study the electrical properties of ZnO, a nanocomposite of ZnO/ PVA has been made [10,11].

2. Experimental section

2.1. Chemical reagents

The chemicals used in this synthesis are Zinc nitrate hexahydrate ($Zn(NO_3)_2 6H_2O$), Sodium hydroxide pellets (NaOH), ethanol (C_2H_5OH), and acetone (C_3H_6O), and Osmunda Regilis leaves extract which has a chemical composition of Hexahydrofarnesyl acetone ($C_{18}H_{36}O$), 2-4-di-t- butyl-phenol ($C_{14}H_{22}O$), and phytol ($C_{20}H_{40}O$). polyvinyl alcohol (C_2H_4O). All the chemicals are purchased from Fisher Scientific and Sigma Aldrich. Ultrapure Milli-Q water is used for washing and making solutions needed in the experiment.

2.2. Instrumentation

An X-ray diffractometer of the Bruker Advance D-8 model has been used to investigate phase identification. It produces Cu-K radiation with a wavelength of 1.5418 Å having a range from 10–80°. The use of a PerkinElmer Spectrum with a scanning range of 4000–400 cm^{-1} validates the presence of functional groups and the names of chemical bonds in the as-synthesized product by the peaks detected. The UV–Vis spectroscopy is conducted using Perkin Elmer750. SEM micrographs are obtained from EVO40.

2.3. Preparation of leaves extract

Garden-fresh Osmunda Regilis (Royal Fern) leaves were extracted from the Delhi Technological University campus, which was firstly washed with tap water and then brushed up using deionized water twice or thrice; after that leaves were dried and then ground. A few amounts of Osmunda Regilis powder is dissolved in a few amount of DI water. The solution was then boiled at 60 °C for 20 min and then it got filtered through Whatman filter paper after filtration it turns brown as shown in the Fig. 1 and then stored in a cold and dry place for further use in the future (see Figs. 2–4).

2.4. Synthesis of ZnO nanorods

In the green route of ZnO NPs by zinc nitrate, 3.51 mL of 0.05 M [$Zn(NO_3)2{\cdot}.6H_2O$] solution was mixed with 38 mL of Osmunda Regilis (Royal Fern) aqueous leaves extract. The mixture was then stirred for 2 h at 400 pm. Then 0.02 M NaOH was added dropwise to reach pH 12 and ultrasonication it stepwise so that all the particles get dispersed. The mixture was again stirred for an extra 1 h at 250 rpm till a solid product with a light yellow color was formed. The precipitate was purified by some re-dispersions from deionized water or ethanol and after that, the product was centrifuged at 6000 rpm for 10 min in two go. The end result was a white powder material that had been dried overnight.

2.5. Preparation of ZnO/PVA thin film

The ZnO /PVA Nanocomposite films were made by the drop-casting method. Synthesized ZnO was added in weight percent ratio by wt% of 5 to PVA from the formula as given below:

$$\mathrm{a}\,(\mathrm{wt}\%) = 100 \times \frac{wz}{wz + wp}$$

where w_z and w_p represent the weight of ZnO and PVA.

The Nanocomposite is produced by dispersing ZnO powder into PVA (2 g) which is dissolved in 50 mL of DI water and then the solution is kept for stirring for 1 h at 80 °C to prevent it from any type of agglomeration. Then films were made through the drop-casting method. Lastly, the films were dried in an oven at 80 °C.

3. Results and discussion

3.1. X-ray diffraction

XRD pattern of synthesized ZnO nanorods which represents the structure of the nanoparticles that we synthesized is crystalline. The keen diffraction peaks remarked at 2θ values which are confirmed from **(JCPDS card no-00-001-1136)** 31.88°, 34.52°, 36.36°, 47.56°, 56.64°, 62.96°, and 66.6°. The peaks are listed as (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (1 1 2), and (2 0 2) diffraction lattice planes which verify the hexagonal wurtzite structure for the ZnO nanorods we synthesized. The crystalline size i.e. t is found using Debye Scherer's formula-.

$$\mathrm{d} = \frac{0.9\lambda}{\beta Cos\theta}$$

where λ is the wavelength of X-ray (1.54 Å), β is the value of Full-width half maxima (radian), θ is Bragg's angle [2dSinθ = n λ].

Sample	d-spacing (nm)	FWHM (radian)	Estimated Crystalline Size (nm)
1.	11.52	0.227	37.4

3.2. U-V Visible spectroscopy

UV–visible spectra of synthesized zinc oxide nanorods are shown in the above figure. The peak is at around 375 nm because of high binding energy at normal/room temperature. Also, we know that by absorption spectroscopy the bandgap increases when

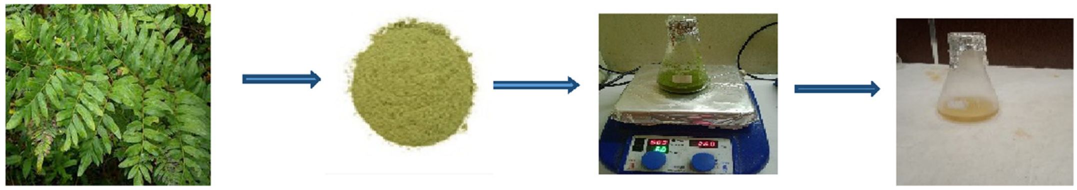

Fig. 1. Schematic diagram representing preparation of plant extract.

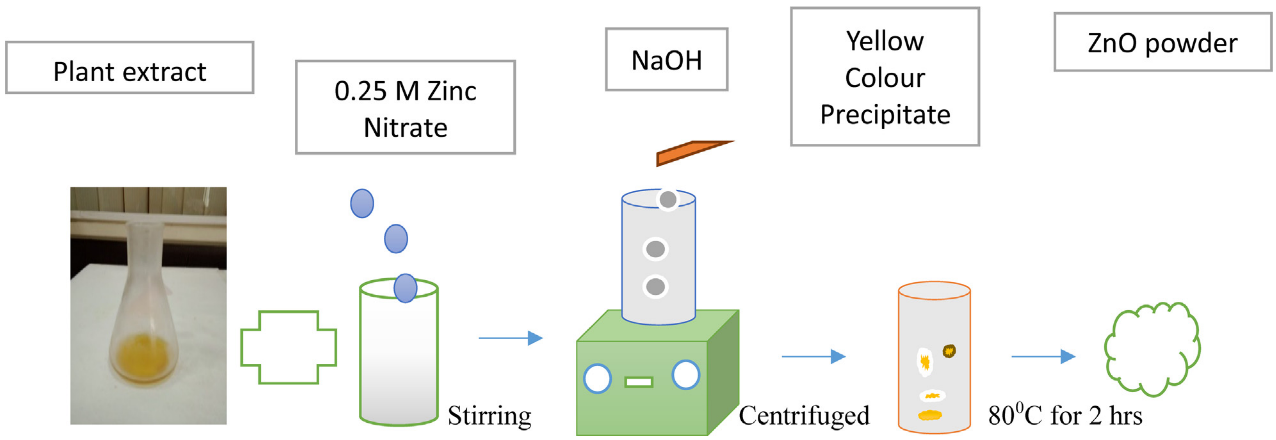

Fig. 2. Schematic representation of the synthesis of ZnO Nanorods.

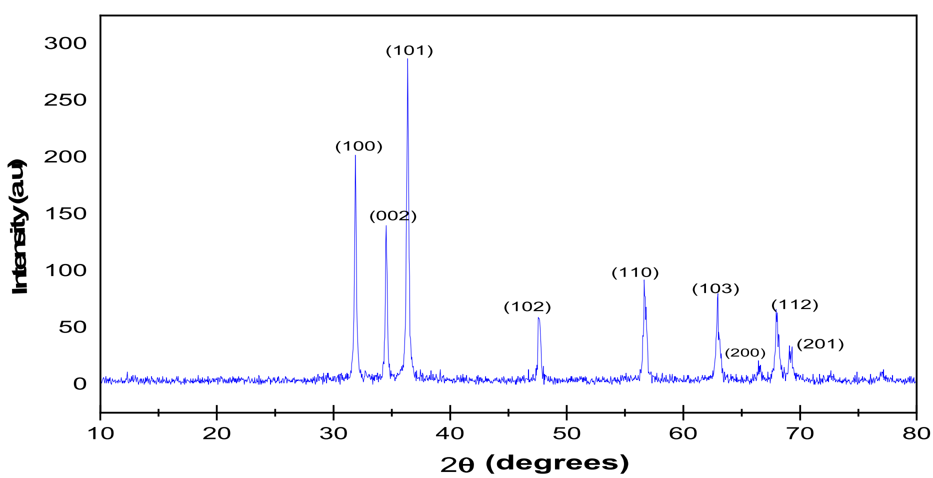

Fig. 3. Graphical depiction of XRD Spectra of prepared ZnO Nanorods.

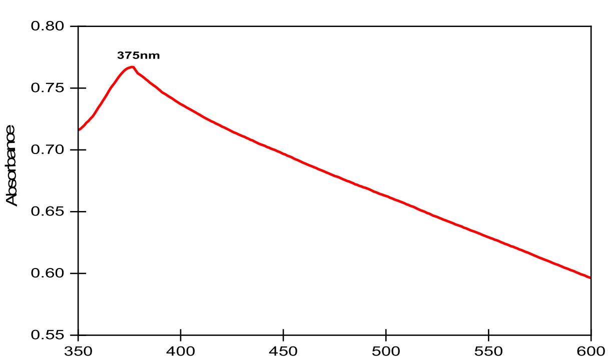

Fig. 4. Graph representing U-V Visible spectra of ZnO nanorods.

the size of the particle decreases as ($t \propto 1/E_g$) where E_g is the bandgap gap of ZnO NPs calculated from the above formula-.

$$(\alpha E)^{1/n} = \beta(E - E_g)$$

where the power factor of the transition mode is n, E_g is the optical bandgap energy, the band tailing parameter is β, and E is calculated from the above formula i.e. E = hν, where h is the Planck's constant, ν is the frequency of UV – Vis spectra. ZnO is a direct bandgap material for allowed transitions therefore n = 1/2.

From the above formula, the bandgap is calculated to be 3.2 eV. Whereas, the actual band gap for ZnO nanorods is found to be 3.8 eV which is larger than the bandgap we found this is how we see that the particle size is inversely proportional to the bandgap because if the particle size is less than it enlarges the energy bandgap.

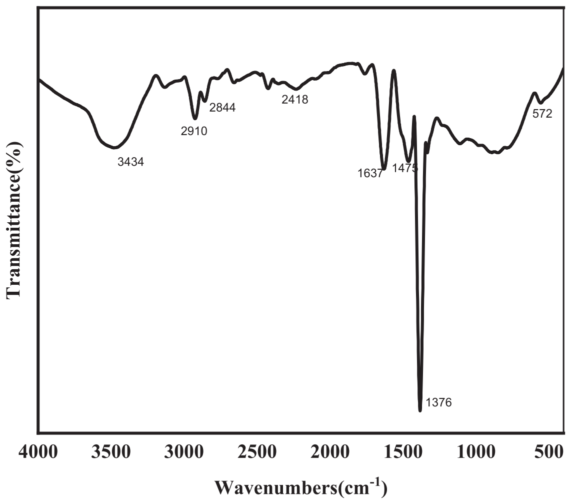

Fig. 5. FTIR spectra of ZnO nanorods.

3.3. Fourier transform infrared spectroscopy

FT-IR studies of Osmunda Regilis aqueous leaves extract and product of synthesized ZnO-NR as shown in Fig. 5. Infrared studies were achieved to find out the purity, nanoparticles nature, and also the proximity of phytochemicals in the material. The phytochemicals such as phenols, amines, alcohols and carboxylic acids can interact with the zinc surface and support in the maintenance of ZnO-NR. The peak at 1196cm^{-1} specifies that it is a C—N stretch which specifies that there is a presence of amines. The peak at 1637 cm^{-1} corresponds to Zn-O stretching and deformation vibration. The peak at 3434 cm^{-1} indicates the OH stretching vibrations which indicate there is the presence of the carboxylic acid functional group.

3.4. Scanning electron microscopy

Fig. 6 shows the SEM images of ZnO Nps, which clearly shows the synthesized ZnO Nanoparticles are nanorods and their size lies between 30 and 100 nm having a crystalline structure, high magnification Fig. 6a and low magnification (6b) SEM images are taken. The image with low magnification shows uniform ZnO nanorods with uniform yield. The image with high magnification nanorods surface is plane. Very minute second particle growth components are interposed (see Figs. 7–8).

3.5. Electrical properties of ZnO/PVA nanocomposite thin films

From the I-V curve, we obtained of ZnO/PVA nanocomposite thin film were measured at normal temperature as given in Figure, we can easily demonstrate that the graph obeys ohms law from its linearity above 0.5 V.

As ZnO represents semiconducting behave as PVA i.e Polyvinyl alcohol represents conducting nature which represents its better charge transport through it [3]. This linear nature in the interface of metal and insulators is very salient in the growth of electronic devices with better performance and trustability.

4. Conclusions

In this exertion, ZnO-NR was synthesized by Osmunda Regilis leaves aqueous solution and Zinc Nitrate, with NaOH as a precursor. The results of XRD committed the regulation of the synthesis procedure which indicates the creation of crystallite zinc Nanorods along with a hexagonal wurtzite structure. UV–Visible spectrum showed a clear peak at 375 nm, from which we determined the value of band gap i.e 3.3 eV. The average size of synthesized ZnO-NR was 37.4 nm. FT-IR studies show the formation of ZnO-NR and contain various phytochemicals which we can determine from peaks at different positions which act as capping and reducing agents for the synthesized ZnO-NR and the I-V characteristics of ZnO/PVA nanocomposite thin film reveal that it is Ohmic nature.

CRediT authorship contribution statement

Parisha Diwan: Experimental work, Methodology, Writing - original draft, Software, Conceptualization, Methodology. **Kanika Sharma:** Conceptualization, Formal analysis, Writing – review &

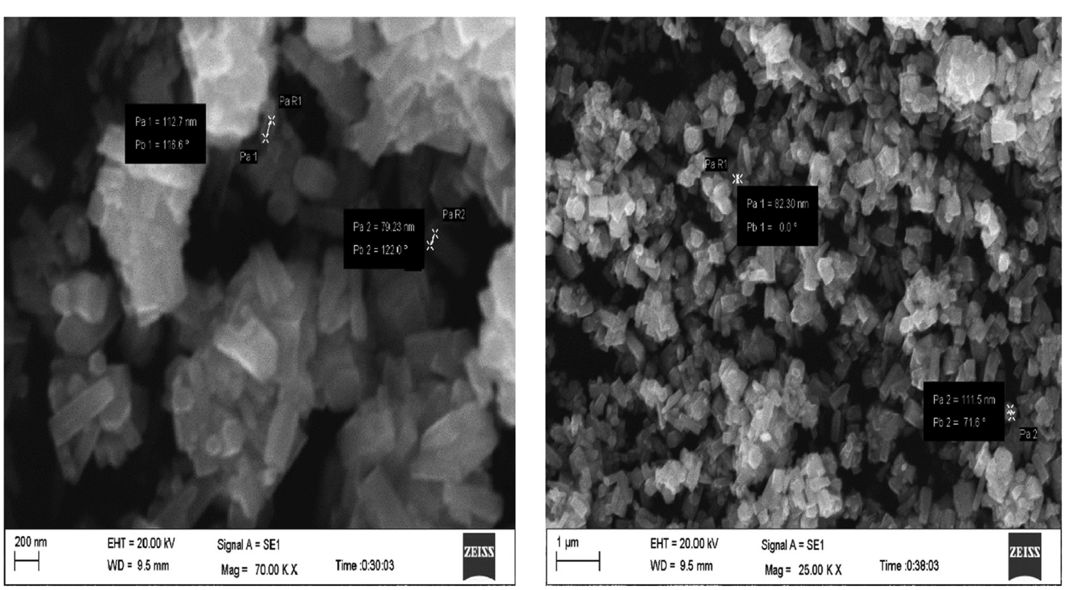

Fig. 6. SEM images of ZnO Nanorods (ZnO-NR) (6a) SEM images showing the particles at a scale of 200 nm and (6b) SEM images showing the particles at a scale of 1 µm 200 nm.

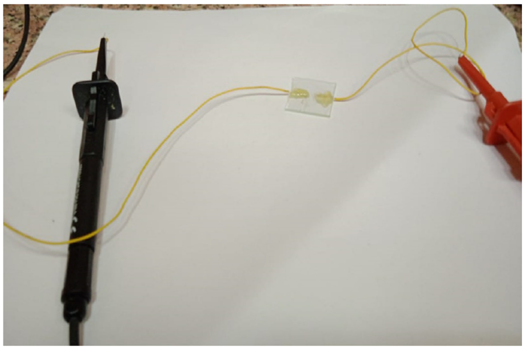

Fig. 7. Electrical measurements of ZnO/PVA thin film.

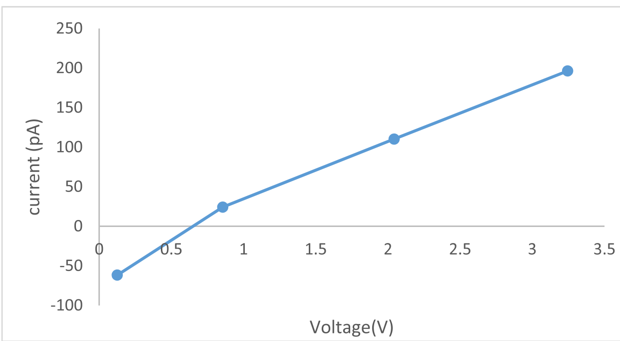

Fig. 8. I-V curve of ZnO-NR/PVA Nanocomposite thin films.

editing, Data curation. **Nitin K. Puri:** Supervision, Project Administration, Resources, Writing – review & editing, Validation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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