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SOL-GEL METHOD BASED SYNTHESIS AND CHARACTERIZATION OF ZNO NANOPARTICLES

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ABSTRACT: In recent times, there has been a growing fascination with semiconductor material nanoparticles due to their distinctive properties and wide-ranging applications. Zinc oxide (ZnO), a semiconductor with a wide band gap (3.37eV), has garnered significant attention in the form of nano-sized particles. In the past decade, ZnO nanoparticles (NPs) have captivated researchers due to their favorable properties and potential applications in optoelectronic devices. This paper presents a study focused on the synthesis and characterization of ZnO nanoparticles using the sol-gel method. In this study, Zinc Oxide (ZnO) nanoparticles (NPs) were synthesized and classified using the sol-gel method. Deionized water served as the solvent, while Zinc Chloride (ZnCl2), Sodium Hydroxide (NaOH), and Zinc Acetate (Zn(CH3CO2)2) were employed as precursors. The prepared NPs were subjected to analysis to investigate their structural, morphological, optical, and magnetic properties. Techniques such as UV-VIS spectroscopy (Ultra Violet visible spectrum), X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) were utilized for this purpose. SEM provided information about the particle size, UV-VIS spectroscopy yielded insights into the band energy of the particles, and XRD was employed to determine the average particle size and lattice contraction percentage of the ZnO NPs sample.

KEYWORDS: Nano Particles, Zinc Oxide (ZnO), sol-gel synthesis, XRD, SEM, UV- VIS spectrum.

I. INTRODUCTION

The demand for the development of nano-sized semiconductors has been steadily rising due to their remarkable optical and electrical properties, which find extensive utilization in the fabrication of nanoscaled electronic and optoelectronic devices with multifunctionality [1].

Zinc Oxide nanoparticles (ZnO NPs) possess a wide range of versatile properties, including high chemical and photostability, low dielectric constant, and efficient UV absorption, making it a highly adaptable material [2]. In recent years, there has been a substantial increase in interest regarding the synthesis and characterization of ZnO NPs to explore their specific properties. ZnO is particularly renowned as an excellent semiconductor material, characterized by its significant bandgap of 3.37 eV, surpassing that of other wide bandgap materials [3]. Moreover, ZnO demonstrates potential in various applications, including dye-sensitized solar cells (DSSCs), medical purposes, and photocatalysis, thereby rendering it an exciting commodity for industrial sectors.

Zinc Oxide (ZnO) exhibits immense promise as a viable option for information storage, optoelectronic applications in shorter wavelengths (UV, green, blue), and sensors due to its resemblance to GaN (gallium nitride). ZnO nanoparticles hold significant potential for a wide range of applications, including solar cells, photocatalysts, photodetectors, biosensors, gas sensors, and nano-generators [4]. From earlier studies it is determined that several techniques are developed for preparing ZnO nanoparticles such as chemical vapor deposition, spray pyrolysis, ultrasonic, microwave-assisted, thermal decomposition, micro-emulsion methods, hydrothermal and precipitation techniques [5]. Many of these

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approaches are not utilized extensively over large scale but chemical synthesis approaches are being utilized broadly because of their low cost and simplicity [6]. When materials are scaled down to the nanoscale, they exhibit unique properties that differ from their bulk counterparts. For instance, alterations in the optical and electrical properties of nanomaterials can lead to significant changes in their structural and mechanical characteristics, as well as an increase or decrease in their chemical properties. These distinctive attributes make nanoparticles (NPs) highly appealing for sensing applications and also pose challenges during their characterization process [7]. So the challenges are lie in determining the correct characterization approach which has minimal capabilities to study the nanomaterial characteristics produced by these approaches [8]. In this current work, the ZnO nanoparticles synthesis by sol-gel technique and ZnO NPs characterization through XRD, SEM and UV-VIS absorbance & photoluminescence spectra, is outlined.

II. LITERATURE SURVEY

H. Musleh et al. [9] presented a research in which ZnO nanoparticles (ZnO NPs) were synthesized using hydrothermal and sol-gel techniques using zinc acetate dihydrate (Zn (CH3COO)2.2H2O) as a row material and methanol as a solvent. The synthesized ZnO NPs showed high purity and revealed a wurtzite (hexagonal) crystal structure with particle size (D) ranged from 25 nm to 28 nm. The UV-VIS absorption spectra of ZnO NPs samples and sensitizing dyes were performed. The experimental results showed a significant efficiency for the fabricated DSSCs of synthesized ZnO NPs via sol gel technique comparing to hydrothermal technique. The EY dye exhibited the best performance among others, where conversion efficiency showed a noteworthy improvement from 0.12 to 1.08 %. Matinise et al.,[10] conducted research and developed a reliable and better process to bio-fabricate ZnO NPs by green technique utilizing the extract Moringa Oleifera as a effective chelating agent. His work concluded that the ZnO nanoparticles size is in between 12.27-30.51 and is synthesized successfully through the extract Moringa Oleifera and characterized by various approaches. Their approach was discussed through the investigations of XRD shed-lighting over nanoparticles polycrystalline behavior. Sutradhar et al., [11] presented ZnO nanoparticles green synthesis by thermal approach and under the radiation of microwave utilizing tomatoes aqueous extract as non-toxic and as eco-friendly reducing material. The authors concluded that the microwave-assisted green synthesis is utilized for preparing zinc oxide NPs. A simple technique was reported utilizing extracts of tomato as a reducing agent to synthesize huge amount of ZnO NPs with well-defied dimensions.

Subramanian Ambika et al. [12] described the synthesis of zinc oxide nanoparticles (ZnO NPs) using Vitex negundo plant extract with zinc nitrate hexahydrate as precursor. Biomolecules present in plant extract were used to hydrolyze metal ions into metal oxide NPs in a single-step green synthesis. The hydrolyzing agents involved the various water soluble plant metabolites such as flavonoid, alkaloids, flavone, phenolic compounds, terpenoids and co-enzymes. C. Jayaseelan et al. [13] preseed a work that described a low-cost, unreported and simple procedure for biosynthesis of zinc oxide nanoparticles (ZnO NPs) using reproducible bacteria, Aeromonas hydrophila as eco-friendly reducing and capping agent. The synthesized ZnO NPs were characterized by a peak at 374 nm in the UV–vis spectrum. Rietveld analysis to

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the X-ray data indicated that ZnO NPs have hexagonal unit cell at crystalline level. The maximum zone of inhibition was observed in the ZnO NPs (25 µg/mL) against Pseudomonas aeruginosa (22 ± 1.8 mm) and Aspergillus flavus (19 ± 1.0 mm). Bacteria-mediated ZnO NPs were synthesized and proved to be a good novel antimicrobial material for the first time in this study.

T. K. Kundu et al [14] reported sol-gel synthesis of ZnO nanoparticles in support of poly (vinayl alcohol) (PVA) molecules. PVA molecules offer plenty of active OH groups and a metal ion-polymer complex is formed via a kind of ligand reaction. The Electron paramagnetic resonance (EPR) spectra of the powders were characterized by a broad resonance peak with an average g=2.0591 owing to presence of defects in the specimens. In comparison, ZnO specimens having micron sized grain which are prepared without using PVA do not show any emission with significant intensity defects play a role in improving the optical emission of ZnO nanoparticles prepared by this method. Mclaren et al., [15] described ZnO crystals photolytic activity and stated that is was better for reducing the particular photo-catalytic activities of specific crystallographic plane (001,101,100) when one can compared its surface area quantitatively. This quantitative analysis is compared with ZnO surfaces photo-catalytic behavior by UHV (Ultra-High Vacuum) techniques. The ZnO fabrication process has exhibited a pronounced effect over shape and size dependent catalytic activities.

III. SYNTHESIS OF ZINC OXIDE NANOPARTICLES

3.1 Synthesis of ZnO nano particles

Sol–gel (SG) process generally undergoes in four stages: salvation, hydrolysis, polymerization and transformation into ZnO solid powder. Zinc chloride (ZnCl2), zinc acetate ($Zn(CH_3\ CO_2\ )_2$) and NaOH, all analytical grades are utilized as precursor to synthesize ZnO nanoparticles. Basically the ZnO nanocrystals production unit contains a reactor with heat. In 100 ml de-ionzied water, 4.23 gm of zinc acetate is dissolved through stirring about 30 minutes. G gm of NaOH pellets are deposited in 100 ml of DI water through stirring for 15 minutes. Drop by drop this solution is added to the prepared Zn acetate solution for achieving pH about 12. Under constant stirring the resulted solution is heated at desired temperature (50°C & 90°C).

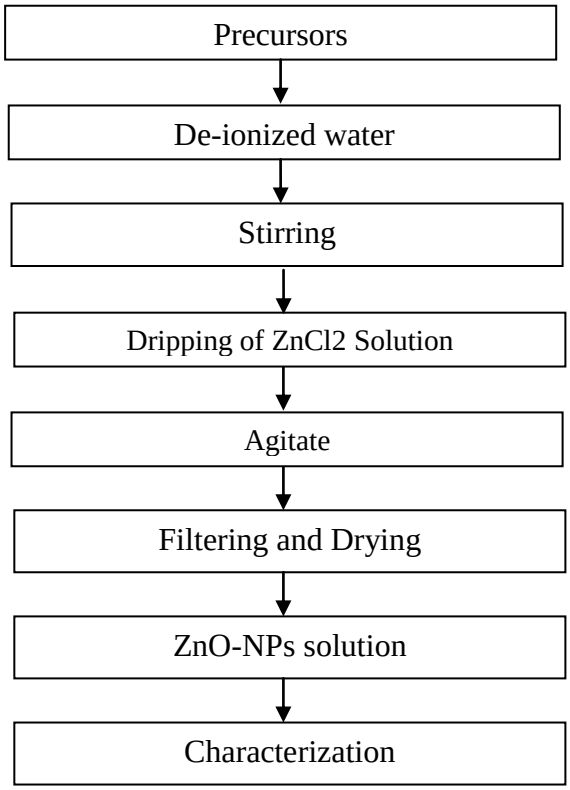

Fig. 1: SOL-GEL SYNTHESIS PROCESS OF ZNO

A 0.5 M of ZnC12 solution is added to the reactor slowly (dripping for 26 to 52 minutes) after gaining required temperature. This process will be conducted under constant stirring and reacting temperature is maintained at desired level. Dripping the ZnC12 solution in aqueous alkaline solution

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leads to ZnO immediate precipitation and suspension color changes from transparent white. After the completion of dripping, it remained is agitated for 2 hours period, maintaining the desired temperature. In the reactor, formed material is filtered, washed several times through de-ionized water, filtered and dried at 70°C maximum temperature in vacuum oven for few hours.

3.2 Synthesis of Na doped ZnO

While preparing the ZnO with 1 wt% Na-doping, an equivalent volume of NaOH is added to the dehydrate solution of Zn acetate. The Na doped zinc oxide synthesis with 1.09gm Zn $(CH3COO)_2 \cdot 2H_2O$ by constant vigorous solution at 70°C in 50 ml methanol solvent for 2 hours. In addition 0.5 M NaOH aqueous solutions is prepared similarly by dissolving 1 gm of NaOH pellet in 50 ml de-ionized water and stirred upto 10 min. This solution acts like a sodium doping source in zinc oxide NPs. After the completion of dissolution, NaOH aqueous solution is dissolved in 0.1M Zn $(CH3COO)_2 \cdot 2H_2O$ aqueous solution by stirring at 70^oC, thus the NaOH is distributed uniformly in $Zn(CH3COO)_2 \cdot 2H_2O$ solution. Then stirred solution initiates white precipitate formation and finally became as gel. This reaction is offered for proceeding at 120 ^oC for 4 hour after the completion of NaOH casting. Next the solution is placed overnight to precipitate settlement. Further this solution supernatant is calcinated for 2hour at 600 ^oC and extracts Na doped zinc oxide NPs.

3.3 Characterization

The dried precipitate is grinded for making the fine powder before utilizing for characterization.

1. Scanning Electron Microscope (SEM): The SEM is a electron microscope type that helps to form a sample surface image through scanning. In the beam the electrons interacted with the surface atoms for generating signals which shed valuable light properties such as electrical conductivity, topography and composition. So named "signals" generated through SEM includes back-scattered and secondary electrons, specimen current, characteristic X-rays and light (because of cathodolumine scence). Basically SEMs have ability of detecting the secondary electrons but it is highly impossible that a single SEM has ability for detecting all of the above mentioned signals. SEM helps for obtaining the specimens high resolution images range from the visible to naked eye to those that are only a few nanometers size.

2. X-Ray Diffraction (XRD): The XRD is a non-destructive kind of analytical approach that gives valuable insight of a crystalline substance lattice structure such as bond angles, unit cell dimensions, etc. The XRD works basing on the constructive interference principle of x-rays and sample concerned must be crystalline. X-rays which are produced by CRT are filtered, collimated and directed to the sample. The interaction which follows produced constructive interference basing on Brag's Law that corresponds to incident radiations wavelength to lattice spacing and diffraction angle. After proper preprocessing the obtained X-rays on diffraction are counted.

3. **UV visible spectrum (UV-VIS):** The NPs size plays a significant role in altering the materials whole properties. Hence semiconductor materials size evaluation has become essential for exploring the material properties. The UV-VIS absorption is broadly utilized method for examining nano-sized particles optical properties. The optical properties of the synthesized ZnO NPs were studied by UV-VIS absorption spectroscopy.

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IV. RESULTS

The Zinc Oxide NPs morphology is examined with the support of Hitachi Model S-300H SEM machine. The ZnO NPs size and structure is studied with Bruker AXS D8 Advance X Ray diffractometer utilizing Cu-Kα radiations that have1.504A.U wavelength. The sample UV visible spectrum is obtained by a Shimatzo's UV visible spectrophotometer-1800. The analysis of UV-ViS is carried out with synthesized samples dilution in de-ionized water (50 vol.% / 50 vol.%), with the readings between the range 200-800nm in double beam spectrometer in 4ml quartz sample holder and solution absorbance is observed.

4.1 X-Ray Diffraction (XRD) Spectrum

The Fig. 2 represents the XRD spectrum of synthesized zinc oxide NPs and confirmed that, structure of wurtzite is hexagonal. The peaks of characteristics (100), (002), (101), (102), (110), (103), (200), (112), and (201) related to ZnO hexagonal structure (JCPDS Card no 01-075-1526) with the preferred orientation along (101) the plane.

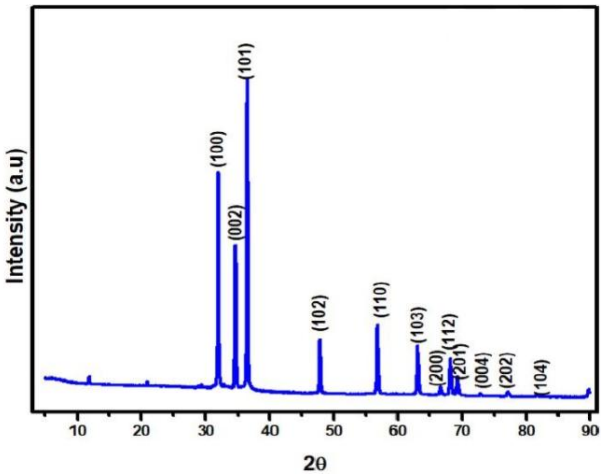

Fig. 2: XRD PATTERN OF ZNO NANOPARTICLES

The ZnO NPs wurtzite structure belongs to the space group (C6V=P63mc) and contains unit cell metrics c = 5.209 Å and a = b = 3.253 Å. The nanaparticles average size is calculated for the peak 101 by Debye-Scherrer formula

$$D = \frac{0.09\lambda}{\beta COS\theta}$$

Where β is full width at half maximum (FWHM), λ is the wavelength of X-ray (1.504 nm) and θ is the angle of Bragg diffraction. The FWHM is measured through Gaussian curve to the higher peak 101. The nanoparticles average size is determined as14.36 nm. The crystallite size calculation results using FWHM (Full Width at Half Maximum) technique from the spectrum of XRD exhibited that for (101) plane the size of the crystal is a function of precursor type and temperature of synthesis. The size of the smallest crystallite is obtained by utilizing chloride as precursor at highest temperature (90^oC) of synthesis.

4.2 Scanning Image Microscopy (SEM)

The samples SEM images are represented in Fig. 3 and 4. Figure (3) show the SEM image of ZnO nanparticles and figure (4) shows the SEM image of sodium doped ZnO nanparticles. From these figures it is clear that the particles are in spherical shape and have granular behavior. The particles agglomeration is observed at higher resolution of SEM image. Agglomeration is caused by aging.

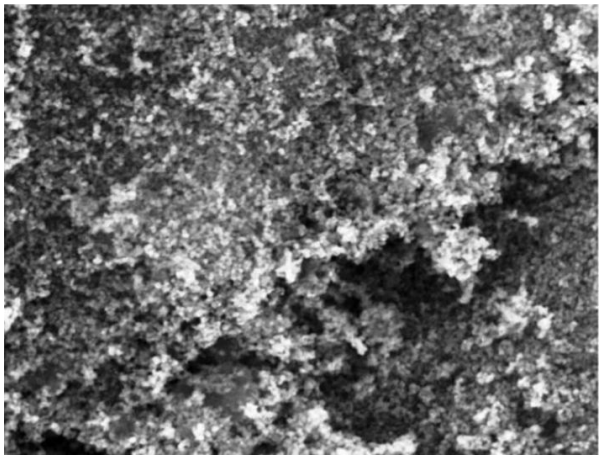

Fig. 3: SEM IMAGE OF ZNO NANO-PARTICLES

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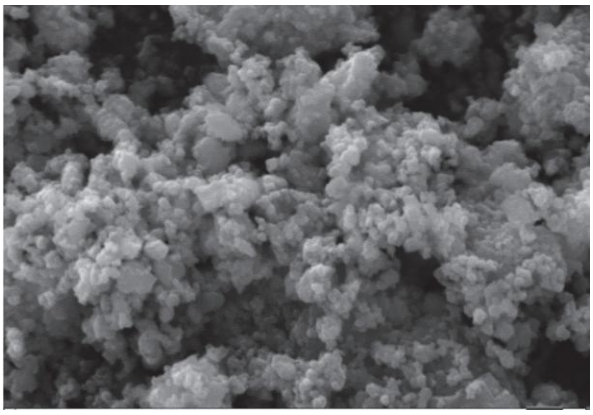

Fig. 4: SEM IMAGE OF NA DOPED ZNO NANO-PARTICLES

4.3 UV Visible Spectrum (UV-VIS)

The Fig. 5 represents sample UV-VIS absorption. In the UV range radiations are absorbed by sample up to 362 nm and most of the visible spectrum radiations are transmitted through ZnO NPs. The Zinc Oxide NPs band gap energy is calculated through extrapolating the curve drawn between (α hν) 2 and (h ν) (represented in Fig. 6).Where α is the coefficient of optical absorption and ν is frequency. The energy of band gap is obtained through exploiting the curve is determined as 3.3 eV.

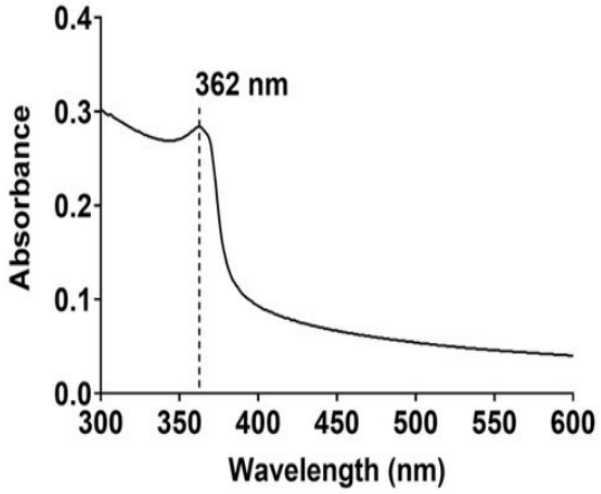

Fig. 5: UV VISIBLE ABSORPTION SPECTRA OF ZNO

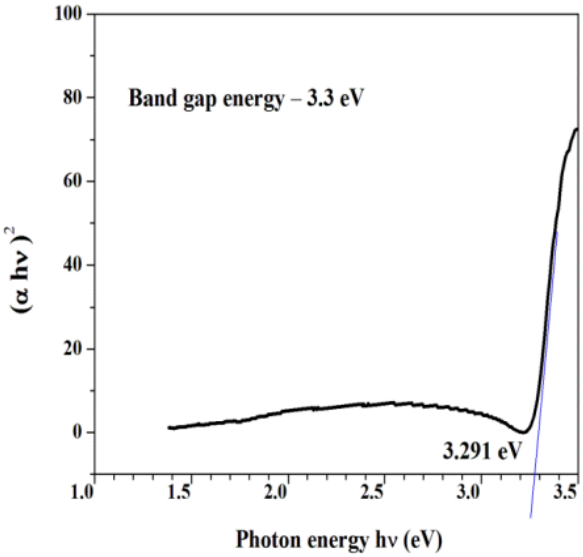

Fig. 6: BAND GAP CURVE OF SYNTHESIZED ZNO NANOPARTICLES

V. CONCLUSION

This research paper focused on the synthesis and characterization of Zinc Oxide (ZnO) nanoparticles using the sol-gel method. The ZnO nanoparticles were prepared and characterized using techniques such as Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and UV-VIS absorption spectroscopy. XRD analysis confirmed the successful production of ZnO nanoparticles with a wurtzite crystal structure, utilizing ZnCl2, $Zn(CH_3\ CO_2\ )_2$, and NaOH as precursors in the sol-gel route. SEM imaging confirmed the formation of spherical granular ZnO nanoparticles. The absorption peak was observed at 362 nm, indicating the characteristic absorption of ZnO nanoparticles. The synthesized nanoparticles exhibited an energy band gap of approximately 3.291 eV. Additionally, the UV-VIS absorption results revealed strong absorption in the UV region (362 nm) for the synthesized samples.

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