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SYNTHESIS AND CHARACTERIZATION OF ZnO NANOPARTICLES

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Abstract

Zinc oxide nanoparticles were successfully synthesized using the co-precipitation method with zinc nitrate and ammonia solution as starting agents. The X-ray diffraction (XRD) analysis, specifically utilizing the full width at half maximum (FWHM) of the peaks, allowed for the determination of an average crystallite size of approximately 28.54 nm, highlighting the nanoscale dimensions of the particles. In addition to XRD, the nanoparticles were characterized through Fourier-transform infrared spectroscopy (FTIR) and ultraviolet (UV) spectroscopy, which provided insights into their chemical composition and optical properties. This comprehensive study offers valuable data on the synthesis and characterization of ZnO nanoparticles, making them promising candidates for applications in nanotechnology, material science and optoelectronics.

Keywords: Zinc oxide; co-precipitation; Optical properties; chemical composition.

1. Introduction

Zinc oxide (ZnO) nanoparticles occupy a prominent posi-tion in the realm of nanomaterial exploration, boasting a myriad of distinctive characteristics and a versatile range of uses across various scientific and industrial fields. Functioning as a semiconductor, ZnO exhibits a broad energy gap, exceptional electrical conductivity, and no-table optical attributes, rendering it a coveted substance for cutting-edge technological progressions [1-3].

ZnO showcase a noteworthy characteristic in the form of size-dependent quantum effects, a phenomenon that gains prominence at the nanoscale [4]. These effects en-dow ZnO with augmented properties, especially in the fields of electronics [5], optics [6], and catalysis [7]. The capacity to govern and fine-tune these properties has spurred investigations into diverse synthesis approaches, with the co-precipitation method emerging as a prominent contender in this exploration. The co-precipitation method is a versatile and widely employed technique for synthesizing ZnO nanoparticles [8]. This approach involves the simultaneous precipita-tion of zinc ions in the presence of a precipitating agent, typically a base. The simplicity, cost-effectiveness, and scalability of the co-precipitation method make it an at-tractive choice for researchers aiming to produce ZnO nanoparticles with tailored characteristics. In the co-precipitation process, zinc ions are introduced to a solu-tion containing the precipitating agent under controlled conditions. The choice of reaction parameters, such as temperature, pH, and concentration, plays a pivotal role in determining the characteristics of the resulting nano-particles [9]. The reaction leads to the formation of ZnO nanoparticles, and the subsequent steps involve careful isolation, washing, and drying to obtain the final product. What sets the co-precipitation method apart is its ability to provide precise control over the size, shape, and crys-talline structure of the synthesized ZnO nanoparticles [10]. The parameters employed during the synthesis pro-cess influence the nucleation and growth of the nanopar-ticles, allowing researchers to tailor the material for spe-cific applications. This fine-tuning is particularly crucial in optimizing the performance of ZnO nanoparticles in electronic devices, sensors, and catalytic processes [11]. The synthesized ZnO nanoparticles are subjected to com-prehensive characterization to unveil their structural, morphological, and optical properties. Techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and UV-Vis spectroscopy are employed in this process. XRD provides insights into the crystalline structure, confirm-ing the wurtzite phase characteristic of ZnO. SEM and TEM offer a closer look at the morphology and size dis-tribution of the nanoparticles, revealing their uniformity and shape. UV-Vis spectroscopy allows researchers to an-alyze the optical properties, such as absorption peaks and bandgap energy, shedding light on the electronic transi-tions within the material. Beyond the synthesis and char-acterization, ZnO nanoparticles synthesized via the co-precipitation method find applications in various techno-logical fronts. In electronics, their unique electrical properties make them suitable for use in sensors, field-effect transistors, and transparent conductive films. The optical properties of ZnO nanoparticles make them valuable in solar cells [12], light-emitting diodes [13], and other optoelectronic devic-

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es. Furthermore, the photocatalytic activity of ZnO nanoparticles has implications in environmental remediation and water purification. In conclusion, the synthesis and characterization of ZnO nanoparticles using the co-precipitation method represent a pivotal avenue in nanomaterial research. This method, with its simplicity and versatility, allows for the precise engineering of ZnO nanoparticles with tailored properties. The exploration of ZnO nanoparticles not only enhances our understanding of nanomaterials but also opens doors to innovative applications across various scientific and technological disciplines, solidifying their position as a cornerstone in the realm of nanoscience and technology.

Materials and Methods:

The experiments utilized zinc nitrate hexahydrate and ammonium solution as essential chemicals. All the reagents employed were of analytical reagent quality and procured from Merck (Mumbai, India). Deionized water was used for the formulation of solutions.

Synthesis of ZnO Nanoparticles

In this synthesis procedure, 2M of Zinc nitrate hexahydrate ($Zn(NO_3)_2 \bullet 6H_2O$) were meticulously dissolved in 100 mL of distilled water at room temperature, resulting in a homogeneous solution. Ammonia solution (NH_4OH) was then carefully added drop wise until a visible precipitation formed. After allowing the reaction to proceed, the precipitate settled at the bottom, and the clear supernatant liquid was decanted. The precipitate underwent thorough washing with ethanol and distilled water to eliminate impurities. Subsequently, it was dried at 80°C overnight to remove any remaining moisture. The final transformation occurred during calcination at 600°C for 2 hours, converting the zinc hydroxide into pure zinc oxide (ZnO) while eliminating organic matter and volatile compounds and the synthesis procedure shown in Fig 1. This meticulously controlled process yielded a purified and analytically suitable zinc oxide sample for further scientific analysis and research applications.

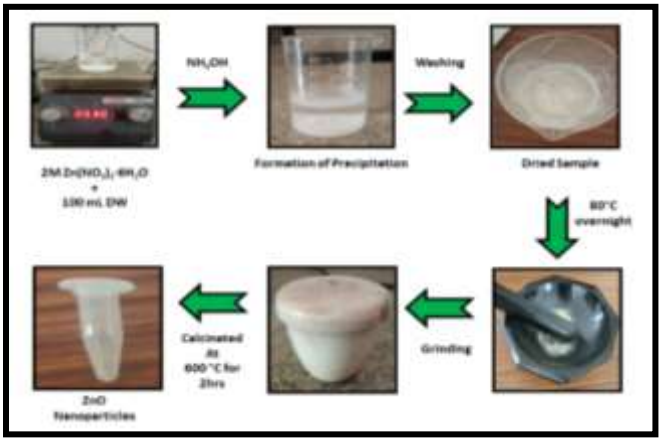

Fig 1: Synthesis procedure of ZnO nanoparticles.

Results and Discussion

X-Ray Diffraction Analysis (XRD):

The X-ray diffraction (XRD) pattern of the synthesized ZnO nanoparticles, as shown in Figure 2, was employed to determine the nature and crystallinity of the sample. By comparing the peak positions in the XRD pattern with the database of the Joint Committee on Powder Diffraction Standards (JCPDS) under reference number 89-0510 [14], it was established that the ZnO nanoparticles possessed a hexagonal crystal system with a primitive lattice structure. The XRD peaks were identified at specific 2θ angles: 31.61°, 34.38°, 36.19°, 47.48°, 56.52°, 62.78°, 66.24°, 67.83°, and 68.96°. These angles correspond to the crystal lattice planes (100), (002), (101), (102), (110), (103), (200), (112), and (201), respectively. Importantly, no additional peaks representing impurities were detected, affirming the purity of the synthesized ZnO nanoparticles.The crystalline size of the ZnO nanoparticles was determined using Debye-Scherrer's equation [15],

$$D=(K \lambda)/(\beta \cos\theta) \ldots\ldots\ldots (nm)$$

Where, relates the crystalline size (D) to the peak width (β), wavelength (λ) of the X-ray source, and the diffraction angle (2θ). This mathematical expression allows for the calculation of the crystallite size, providing valuable insights into the structural characteristics of the nanoparticles. The average crystalline size of ZnO nanoparticles was found to be 26.71 nm.

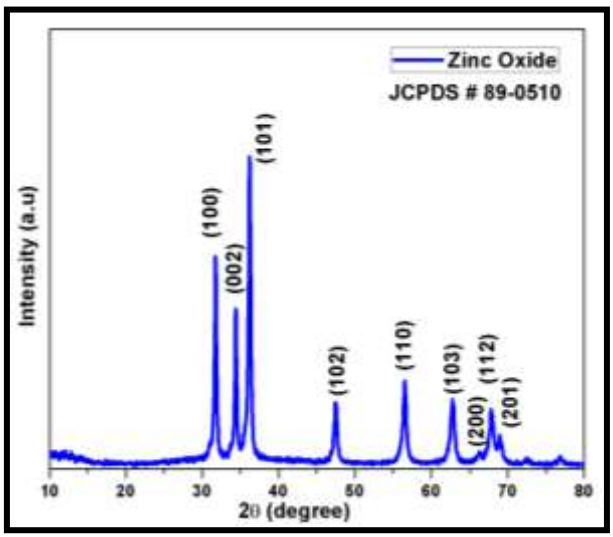

Fig 2: XRD diffraction pattern of ZnO nanoparticles

2 theta (degree)		d Spacing(A°)		(hkl)	FWHM (deg)	Crystallite Size D (nm)	Micro strain
Observed	Standard	Observed	standard				
31.73	31.77	2.8177	2.8135	(100)	0.2481	33.2751	0.2182
34.38	34.43	2.6064	2.6027	(002)	0.2222	37.4096	0.1796
36.21	36.26	2.4787	2.4751	(101)	0.3113	26.8383	0.2380
47.48	47.55	1.9133	1.9105	(102)	0.3887	22.3184	0.2209
56.52	56.61	1.6268	1.6244	(110)	0.4512	19.982	0.2098
62.78	62.87	1.4788	1.4768	(103)	0.4692	19.8265	0.1922
66.28	66.40	1.4090	1.4067	(200)	0.3708	25.5764	0.1420
67.82	67.97	1.3807	1.3780	(112)	0.4859	19.6925	0.1807
68.95	69.11	1.3608	1.3580	(201)	0.2711	35.53	0.0987

Table 1: Data corresponding to ZnO nanoparticles

FTIR(Fourier Transform Infrared Spectroscopy)

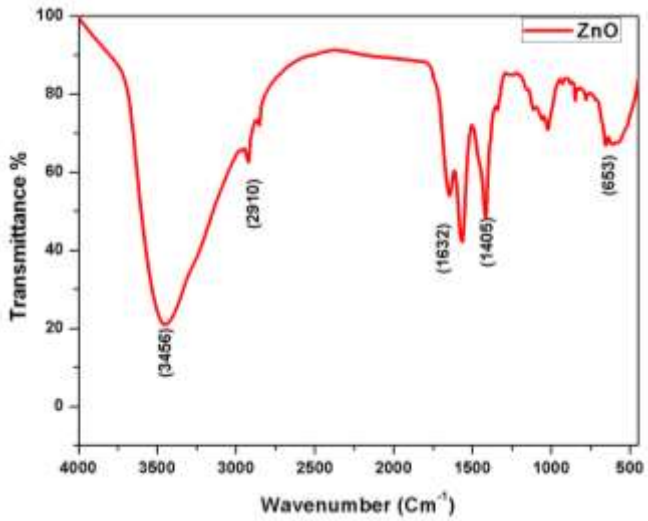

Fig 3: FTIR spectrum of ZnO nanoparticle

The Fourier Transform Infrared Spectroscopy (FTIR) spectrum of ZnO nanoparticles reveals in Fig 3 which has distinctive peaks at wavenumbers 3456, 2910, 1632, 1405, and 653 cm-1. The peak at 3456 cm-1 [16] suggests the presence of O-H stretching vibrations, indicative of hy-droxyl groups or water molecules, common in metal ox-ides. The peak at 2910 cm-1 corresponds to the asymmet-ric stretching vibration of C-H bonds, potentially signal-ing the presence of organic contaminants. The 1632 cm-1 peak is likely associated with bending vibrations of water or hydroxyl groups, as well as C=O [17] stretching vibrations in carbonyl groups. The 1405 cm-1 peak may be linked to the symmetric stretching vibra-tions of carboxylate ions or nitrate groups, suggesting the presence of organic acids or salts. Finally, the 653 cm-1 peak is characteristic of the bending vibrations of Zn-O bonds in zinc oxide [18], confirming the nanoparticle composition. Careful consideration of synthesis conditions and reference spectra is advisable for a more precise interpretation of the FTIR results.

UV-Visible Spectroscopy:

The UV-Vis spectrum of zinc oxide (ZnO) nanoparticles shown in Fig 4 (a) which exhibits distinct absorption characteristics, displaying a notable decrease in absorption between 200 and 300 nm, indicative of relative transparency in this wavelength range. Beyond 300 nm, there is a discernible increase in absorption, culminating in a pronounced peak at 441 nm. This maximum absorption at 441 nm suggests efficient light absorption by ZnO nanoparticles in the visible spectrum. Moving on to the bandgap, the observed value of 3.7 eV underscores the semiconductor nature of ZnO shown in Fig 4 (b). This bandgap represents the energy difference between the valence and conduction bands, influencing the material's electronic and optical properties. A bandgap of 3.7 eV implies a moderate energy threshold for electron transitions, affecting the absorption onset and contributing to the observed peak at 441 nm in the UV-Vis spectrum [19]. The intricate interplay between absorption features and bandgap in ZnO nanoparticles is pivotal for tailoring their properties for various applications, such as in optoelectronics and sensors, where the bandgap serves as a crucial parameter for optimizing performance.

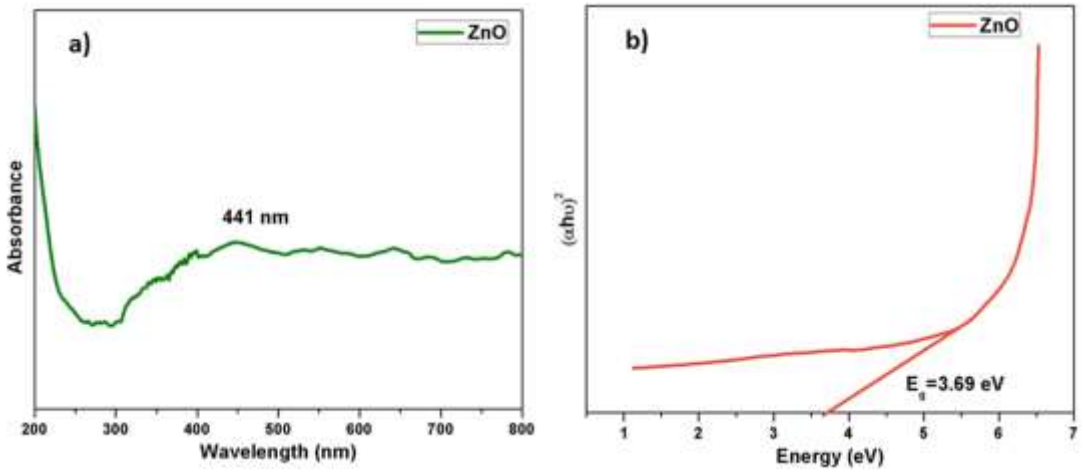

Fig 4: a) UV-Vis spectrum and b) Tauc plot of ZnO nanoparticles

Conclusion:

In summary, the present study has successfully synthesized and characterized zinc oxide (ZnO) nanoparticles, offering valuable insights into both their structural and optical features. X-ray diffraction (XRD) analysis unveiled a well-defined hexagonal crystal system, showcasing the precision of the employed synthesis method and revealing a calculated crystallite size of 26 nm. The Fourier-transform infrared (FTIR) spectroscopy results provided essential chemical characterization by confirming the presence of metal oxide bonds within the nanoparticles, complementing the structural understanding obtained from XRD. The UV studies contributed to a deeper understanding of the material's optical behavior, exposing a bandgap of 3.69 eV, a critical parameter influencing electronic properties. This thorough characterization sets the stage for the tailored utilization of these ZnO nanoparticles across a spectrum of technological applications, spanning from optoelectronics to catalysis, leveraging their distinctive structural and optical attributes.

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