CONTROLLED SYNTHESIS OF ZnO NANOPARTICLES FROM ZINC ACETATE DIHYDRATE: INSIGHTS INTO STRUCTURAL PROPERTIES

ILAHA V. IZZATOVA
Institute of Radiation Problems of Ministry of Science and Education
AZ1143, B.Vahabzade 9, Baku, Azerbaijan

This study aims to synthesize ZnO nanoparticles from zinc acetate dihydrate and investigate the effects of different solvothermal treatment conditions on their structural and morphological properties. ZnO nanoparticles were initially prepared as nanodots through a solution-based method, followed by a solvothermal treatment to promote recrystallization into more structured nanoparticles. The synthesized materials were characterized using X-ray diffraction (XRD) to assess their crystalline phases, and the effects of varying temperatures (150 °C and 200 °C) and treatment durations (24 h and 120 h) were evaluated. The XRD analysis confirmed the formation of wurtzite-type ZnO nanoparticles, with changes in diffraction peak intensities and widths indicating the effects of solvothermal treatment on crystallinity and morphology. Notably, higher temperatures and longer treatment times resulted in nanoparticles with enhanced crystallinity and anisotropic shapes. The successful synthesis of ZnO nanoparticles with controlled morphological characteristics through solvothermal methods opens new avenues for their application in advanced technologies.

Keywords: ZnO nanoparticles, X-ray diffraction, nanostructures, nanocrystalline.
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1. INTRODUCTION

Zinc oxide (ZnO) nanoparticles have garnered significant attention in recent years due to their remarkable optical, electronic, and catalytic properties, which are largely influenced by their size and morphology [1-4]. As a wide-bandgap semiconductor with a bandgap of approximately 3.37 eV, ZnO exhibits unique characteristics that make it a valuable material for various applications across multiple fields, including electronics, photonics, environmental remediation, and biomedicine. The increasing demand for nanomaterials in advanced technologies has spurred extensive research into the synthesis and functionalization of ZnO nanoparticles to enhance their properties and broaden their applicability. The unique optical properties of ZnO nanoparticles are attributed to quantum confinement effects, which allow for tunable photoluminescence and high transparency in the visible spectrum [5, 6]. This makes ZnO nanoparticles particularly appealing for use in optoelectronic devices, such as light-emitting diodes (LEDs) and solar cells. Furthermore, their photocatalytic activity has established ZnO as an efficient catalyst for the degradation of organic pollutants, contributing to advancements in environmental remediation technologies. In addition to their optical and electronic applications, ZnO nanoparticles exhibit excellent antimicrobial properties, making them suitable for incorporation into drug delivery systems and various biomedical applications [7-11]. Their biocompatibility and ability to be functionalized with therapeutic agents allow for targeted drug delivery and controlled release, enhancing treatment efficacy while minimizing side effects.

The synthesis of ZnO nanoparticles can be achieved through various methods, including sol-gel processes, hydrothermal synthesis, and chemical vapor deposition. Among these, the sol-gel method using zinc acetate dihydrate as a precursor is widely recognized for its simplicity and ability to produce high-purity nanoparticles. This method allows for precise control over particle size and morphology, which are crucial for optimizing the nanoparticles' properties for specific applications. This article provides a comprehensive overview of the synthesis of ZnO nanoparticles using zinc acetate dihydrate as a precursor, focusing on the detailed preparation process, optimization strategies, and characterization techniques. By elucidating these aspects, we aim to contribute to the growing body of knowledge surrounding ZnO nanoparticles and their potential applications in various fields, ultimately fostering further research and innovation in nanotechnology. Our future research will focus on investigating the neutron transmutation effects on high-purity ZnO nanoparticles. The purity of the nano ZnO sample will be a critical factor in ensuring accurate and reliable results [12-16]. We will carefully control the synthesis process to minimize impurities and use advanced characterization techniques, such as XRD, EDX, and INAA, to verify the material's purity. By studying the neutron interaction with pristine ZnO nanoparticles, we aim to gain deeper insights into the transmutation process and its potential applications in various fields.

2. SYNTHESIS AND CHARACTERIZATION OF ZNO NANOPARTICLES

Zinc oxide (ZnO) nanoparticles were synthesized using zinc acetate dehydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$, ACS 98.0–101.0 %, Alpha Aesar) as the precursor material. A precise quantity of 32.9 g of zinc acetate dihydrate was dissolved in 0.5l of absolute ethanol within a round-bottom three-neck glass flask equipped with a distillation apparatus. The mixture was subjected to continuous stirring and heated to 80 °C. During this heating process, approximately 0.3l of condensate was collected and discarded, concentrating the precursor solution. After the heating stage, the remaining solution in the glass flask was rapidly

131, H.Javid ave, AZ-1073, Baku
Institute of Physics
E-mail: jophphysics@gmail.com

cooled to room temperature. To restore the initial volume to 0.5l, an additional 0.3l of absolute ethanol was added. Subsequently, 5 g of lithium hydroxide (LiOH, ≥98 %, Sigma-Aldrich) was incorporated into the mixture and thoroughly dissolved using an ultrasonic bath, which aided in the formation of ZnO nanoparticles. To isolate the ZnO nanoparticles from the suspension, the solution was centrifuged at 10,000 rpm for 20 minutes. The sediment containing the nanoparticles was then washed five times with absolute ethanol to eliminate any residual unreacted precursors or byproducts. To prevent agglomeration of the nanoparticles, 5 mL of Milli-Q water was added to the collected sediment, which was subsequently frozen at −80 °C. The resulting product underwent freeze-drying to obtain a stable powder of ZnO nanoparticles. In addition to nanoparticles, ZnO nanoparticles were synthesized through solvothermal treatment of the unwashed ethanolic suspension of ZnO nanoparticles. 40 ml of this suspension was placed in Teflon-lined stainless steel autoclaves (Parr Acid Digestion Bombs, Parr Instrument Company, USA), filled to 50% capacity. The autoclaves were then sealed and subjected to thermal treatment in an oven at two different temperatures, 150 °C and 200 °C, for varying durations of 20 hours and 120 hours. Upon completion of the solvothermal treatment, the nanoparticles were separated from the suspensions by centrifugation, followed by washing and freeze-drying, mirroring the procedure used for the ZnO nanoparticles. This method ensured the formation of distinct rod-like nanoparticles from the initial nanoparticle suspension.

All synthesized samples (ZnO nanoparticles) were characterized using X-ray diffraction (XRD) to assess their crystallinity and phase composition. The XRD analysis was conducted on an X'Pert PRO high-resolution X-ray diffractometer (PANanalytical B.V., Netherlands). The diffraction patterns were collected over a 2θ range of 30° to 60°, with a step size of 0.034° per 100 seconds using a fully opened X'Celerator detector and CuKα radiation. The obtained XRD patterns were analyzed and resolved using the JCPDS database (International Centre for Diffraction Data, PA, USA) to confirm the presence of the wurtzite crystal structure of ZnO.

3. RESULTS AND DISCUSSION

The synthesized Zinc Oxide (ZnO) nanoparticles, alongside their recrystallized forms as nanoparticles, were systematically analyzed to elucidate their structural properties. The preparation involved manipulating various recrystallization parameters, specifically varying the time (20 and 120 hours) and temperature (150 °C and 200 °C) during solvothermal treatment. The initial characterization of these nanoparticles was conducted using X-ray diffraction (XRD), a critical technique for determining the crystalline structure of materials. The XRD patterns obtained for all samples exhibited characteristic peaks corresponding to the wurtzite crystal structure of ZnO, as confirmed by the Joint Committee on Powder Diffraction Standards (JCPDS 01-089-1397) (Fig. 1a–e).

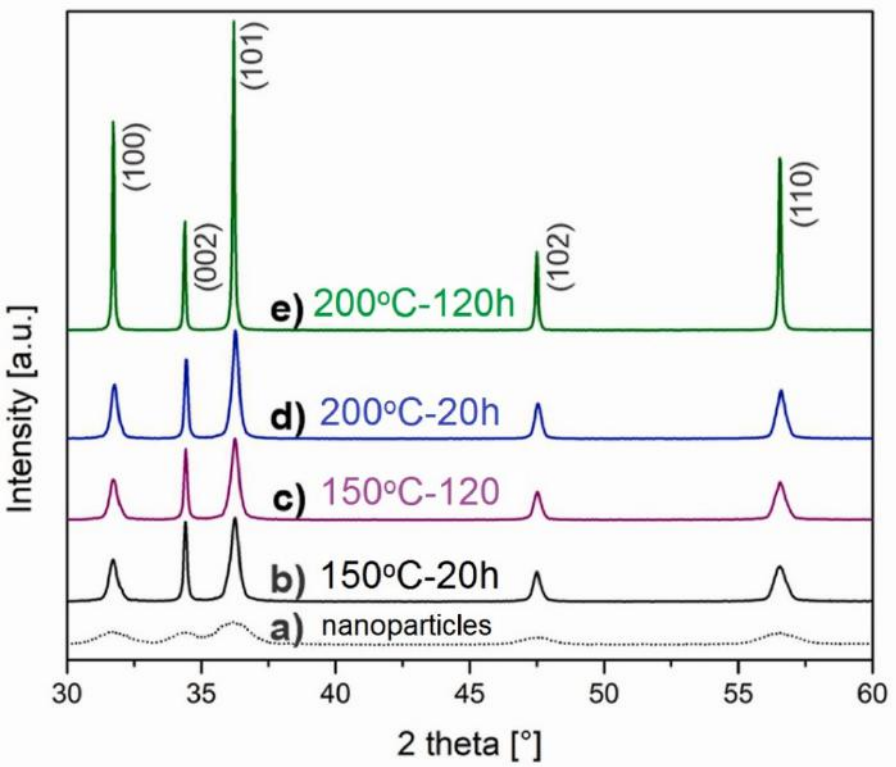

Fig. 1. X-ray Diffraction (XRD) patterns of ZnO nanoparticles synthesized under varying solvothermal conditions: (a) ZnO nanoparticles, (b) recrystallized nanoparticles at 150 °C for 20 h, (c) recrystallized nanoparticles at 150 °C for 120 h, (d) recrystallized nanoparticles at 200 °C for 20 h, and (e) recrystallized nanoparticles at 200 °C for 120 h.

This structure is known for its hexagonal symmetry and is one of the most stable forms of ZnO at room temperature. The diffraction pattern for the synthesized ZnO nanoparticles revealed broad maxima (Fig. 1a), indicative of nanosized crystals. Such broadening is typically associated with smaller crystallite sizes, which in this case confirmed the formation of nanoparticles through the employed synthesis method. From the XRD analysis, we resolved a uniform size distribution of approximately 5 nm for the ZnO nanoparticles, corroborating their crystalline nature as illustrated in Fig. 1a-e. The narrow particle size distribution is significant as it suggests uniformity in the synthesis process, which is crucial for applications where consistent properties are required. The structural features observed in the nanoparticles align well with documented characteristics in the literature, highlighting the reproducibility and reliability of the synthetic approach employed [17]. Following the initial synthesis, the ZnO nanoparticles underwent solvothermal treatment, a process designed to enhance their crystallinity and promote the growth of nanoparticles. This method involves dissolving the nanoparticles in a solvent and subjecting them to elevated temperatures and pressures, which facilitates particle growth and reshaping. The XRD patterns for the solvothermally treated samples (Fig. 1b–e) displayed pronounced changes, with narrowed and intensified diffraction peaks. These changes are indicative of improved crystallinity and structural ordering within the ZnO nanoparticles, a transformation that is beneficial for enhancing their optical and electronic properties.

A notable observation from the XRD analysis was the trend in the relative intensity of the (002) reflection. As treatment temperature and duration increased, the intensity of this particular reflection decreased, culminating in the weakest intensity observed for the sample treated at 200 °C for 120 hours (Fig. 1e). This decrease suggests a shift towards the prevalence of non-polar facets within the ZnO crystal structure, indicating that the high-energy environment fostered by the solvothermal conditions encourages the development of anisotropic, rod-like morphologies. This phenomenon aligns with prior findings that highlight the influence of processing conditions on the crystallographic facets present in nanostructured materials [18, 19]. In examining the relationship between particle morphology and XRD patterns, it was observed that samples exhibiting elongated dimensions along the (001) plane displayed enhanced peak intensities corresponding to these specific planes. This observation underscores the growth orientation of the nanoparticles and suggests that the solvothermal treatment selectively favors the growth of specific crystallographic directions, leading to anisotropic particle shapes that are crucial for various applications. Conversely, it is important to recognize the effects of preferred orientation on the interpretation of diffraction patterns. Rod-like structures, particularly when they exhibit a high aspect ratio (length-to-width ratio), tend to align in a manner that emphasizes certain crystal planes during XRD analysis. This preferred orientation can lead to diffraction patterns dominated by orthogonal planes, complicating the interpretation of the overall shape and crystallinity of the ZnO powder. As a result, accurate conclusions regarding the shape and size distribution may be hindered, necessitating careful consideration during structural analysis. The implications of these structural transformations are profound, particularly for applications in fields such as photocatalysis, sensors, and optoelectronics. The transition from nanoparticles to nanoparticles not only enhances the surface area of the ZnO nanoparticles but also optimizes their optical and electronic properties. Increased surface area facilitates greater interaction with surrounding media, which is advantageous for photocatalytic applications, where enhanced reactivity is desired. Additionally, the specific crystallographic orientations of the nanoparticles may provide improved charge carrier dynamics, essential for the performance of optoelectronic devices. In conclusion, the detailed analysis of the synthesized ZnO nanoparticles and their transformation into nanoparticles provides critical insights into the relationships between synthesis parameters and the resultant structural characteristics. The findings contribute to the understanding of the processing-structure-property relationships in ZnO nanoparticles and underscore the potential for tailoring their properties for diverse applications. Future work should focus on further elucidating the mechanisms underlying the structural evolution during synthesis, as well as exploring the functional performance of the ZnO nanostructures in targeted applications such as environmental remediation and advanced electronic devices.

CONCLUSION

In this study, ZnO nanoparticles were successfully synthesized using zinc acetate dihydrate as a precursor through a two-step process involving the preparation of nanodots followed by solvothermal treatment. The structural analysis conducted via X-ray diffraction (XRD) confirmed the formation of wurtzite-type ZnO, demonstrating that the nanoparticles exhibited distinct characteristics dependent on the temperature and duration of the solvothermal treatment. The findings indicate that higher temperatures and longer treatment times lead to the formation of more crystalline nanoparticles with anisotropic shapes. The ability to tailor the morphology and crystallinity of ZnO nanoparticles through such methods holds significant potential for enhancing their applications in various fields, including photocatalysis, sensors, and electronic devices. Future research will focus on optimizing these synthesis parameters to further improve the performance of ZnO nanoparticles for specific applications.

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