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Synthesis and Structural Characterizations of ZnO @ NiO Nanocomposite

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Abstract

Nanocomposites offer a wide range of versatile applications in the fields of nanotechnology and material science due to their unique combination of properties arising from the synergistic interaction between different nanoscale components. In this study, a ZnO@NiO nanocomposite was successfully synthesized using the co-precipitation method and its structural properties were comprehensively characterized. The surface morphology analyzed through scanning electron microscopy (SEM) revealed a highly porous and agglomerated structure with embedded rod-like and plate-like crystallites, indicating good interfacial interaction between ZnO and NiO phases. Energy-dispersive X-ray spectroscopy (EDX) confirmed the elemental composition, with nickel as the dominant element (77.48 at. %), followed by zinc (12.56 at. %) and oxygen (9.96 at. %), supporting the formation of a NiO-rich composite. The X-ray diffraction (XRD) pattern showed distinct and sharp peaks corresponding to hexagonal ZnO (JCPDS Card No. 36-1451) and cubic NiO (JCPDS Card No. 47-1049), with prominent reflections from ZnO planes such as (100), (002), and (101), and NiO planes like (111), (200), and (220), confirming the high crystallinity and phase purity of the nanocomposite. The successful integration of ZnO and NiO at the nanoscale with well-defined crystal structures suggests potential for applications in gas sensing, catalysis, and electronic devices.

Keywords: Nanocomposites, Synergistic, Agglomerated, Crystallinity, Electronic Devices

I. Introduction:

Nanocomposites of metal oxide semiconductors play a pivotal role in advancing the frontiers of nanotechnology and material science, owing to their remarkable structural, electrical, optical, and catalytic properties [1, 2]. By combining two or more metal oxide semiconductors at the nanoscale, these composites exhibit synergistic effects that significantly enhance their functional performance compared to individual components [3, 4]. Their tunable band gaps, large specific surface areas, and strong interfacial interactions make them ideal candidates for a variety of applications such as gas sensors, photocatalysts, solar cells, supercapacitors, and electronic devices [5, 6]. For instance, heterojunctions formed between different metal oxides like ZnO and NiO facilitate efficient charge separation and transport, which is crucial in improving sensitivity and selectivity in gas sensing or increasing photocatalytic efficiency under visible light [7, 8]. The thermal and chemical stability of metal oxide nanocomposites allows them to perform reliably under harsh environmental conditions. Their versatility,

combined with the ability to tailor their properties through doping, morphology control, and surface modification, positions metal oxide semiconductor nanocomposites at the forefront of next-generation smart materials [6-9].

Zinc oxide (ZnO) is a widely studied n-type semiconductor material that exhibits a unique combination of optical, electrical, and chemical properties, making it highly valuable across diverse scientific and industrial domains [10, 11]. It crystallizes in a hexagonal wurtzite structure and possesses a wide direct band gap of approximately 3.37 eV, along with a high exciton binding energy (~60 meV), which contributes to its strong ultraviolet (UV) absorption and photoluminescence properties. ZnO is well-known for its excellent chemical stability, non-toxicity, and biocompatibility, making it suitable for applications in electronics, optoelectronics, photovoltaics, gas sensing, and biomedical devices. Its large surface area and high electron mobility enhance its sensitivity in sensing applications, while its photocatalytic capabilities under UV light make it effective for environmental remediation and self-cleaning surfaces [12, 13]. Furthermore, ZnO's piezoelectric and pyroelectric properties enable its use in nanogenerators and sensors. Due to its ability to be synthesized in various nanostructured forms such as nanorods, nanowires, and nanoparticles [14, 15]. ZnO remains a material of choice in nanotechnology and material science research.

Nickel oxide (NiO) is a p-type semiconductor material that has garnered significant attention in nanotechnology and material science due to its excellent chemical, thermal, and electrochemical properties [16, 17]. It crystallizes in a cubic rock-salt structure and typically exhibits a wide band gap ranging from 3.6 to 4.0 eV, depending on the synthesis method and material stoichiometry. NiO is known for its strong electrical resistivity, high thermal stability, and good catalytic activity, making it ideal for applications in gas sensors, supercapacitors, photocatalysis, batteries, and electrochromic devices [17-19]. As a p-type material, NiO is particularly valuable when paired with n-type semiconductors (such as ZnO) to form p–n junctions, which enhance charge separation and improve performance in optoelectronic and sensing devices [20, 21]. The surface of NiO can be easily modified through doping or nanostructuring to fine-tune its properties for specific applications. NiO is also environmentally friendly and cost-effective, further supporting its widespread use in sustainable technologies [21-23]. The aim of this research paper is to synthesize and structurally characterize a ZnO@NiO nanocomposite using the co-precipitation method and to explore its potential as a multifunctional material in the field of nanotechnology. By combining the distinctive properties of n-type ZnO and p-type NiO semiconductors, the study seeks to develop a heterostructured nanocomposite with enhanced structural integrity and interfacial interaction. Through detailed morphological, elemental, and crystallographic analyses using SEM, EDX, and XRD techniques, the research aims to confirm the successful formation of the ZnO@NiO composite and to understand its structural properties at the nanoscale.

II. Materials and Methods:

In this study, the ZnO@NiO nanocomposite was synthesized using the co-precipitation method. Zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$) and nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$) were used as the precursors for zinc oxide (ZnO) and nickel oxide (NiO), respectively. Sodium hydroxide (NaOH) was employed as a precipitating agent to initiate the formation of the composite. The process began by preparing aqueous solutions of zinc and nickel nitrates in the desired stoichiometric ratios. Sodium hydroxide solution was then added dropwise to the mixed solution under continuous stirring,

maintaining a pH of 10–11. The resulting precipitate was allowed to age for 24 hours at room temperature, followed by filtration and washing with deionized water to remove any excess ions. The precipitate was dried at 200°C for 6 hours and subsequently annealed at 400°C for 2 hours to form the ZnO@NiO nanocomposite [24, 25].

III. Result and Discussion:

Figure 1 shows the scanning electron microscopy (SEM) micrograph of the synthesized ZnO@NiO nanocomposite at a magnification of 10,000x.

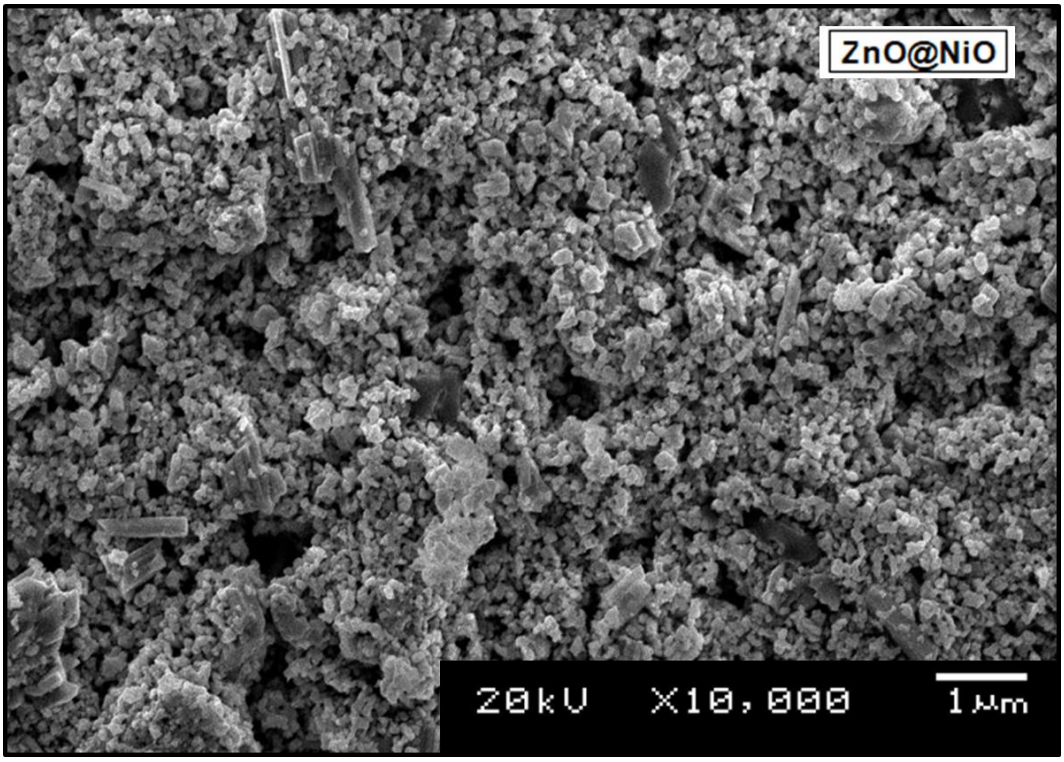

Fig. 1. SEM micrograph of ZnO@NiO nanocomposite

The image reveals a highly porous and agglomerated surface morphology, characteristic of nanoscale materials synthesized via the co-precipitation route. The composite consists of fine, irregularly shaped nanoparticles forming a dense network, which suggests a large surface area beneficial for applications such as catalysis and gas sensing. Figure 1 also shows the rod-like and plate-like structures are sporadically embedded within the nanoparticulate matrix, likely indicating the presence of ZnO crystallites decorated with NiO nanostructures [25, 26]. The heterogeneous distribution and intimate contact between ZnO and NiO phases contribute to the formation of a well-integrated nanocomposite, which may enhance charge separation and improve functional performance in potential applications. The observed microstructure aligns well with the expected features of a binary metal oxide composite, confirming successful synthesis and morphological integration [26, 27].

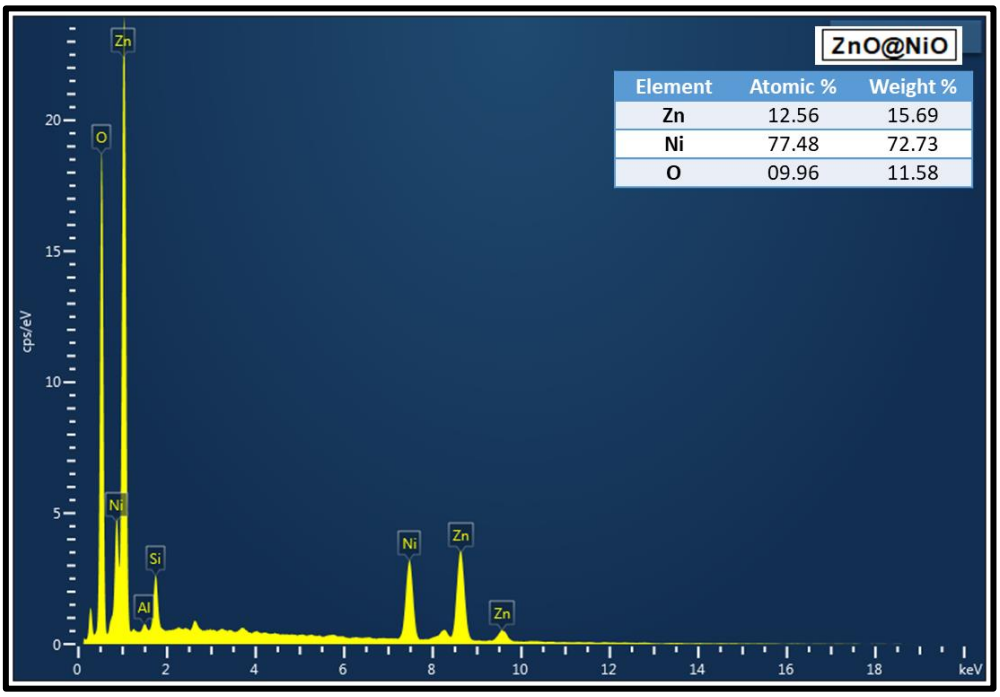

Fig. 2. EDX spectrum of ZnO@NiO nanocomposite

Figure 2 presents the energy-dispersive X-ray (EDX) spectrum of the ZnO@NiO nanocomposite, confirming the elemental composition of the synthesized material. The spectrum displays prominent peaks corresponding to zinc (Zn), nickel (Ni), and oxygen (O), which are the key constituents of the composite. Quantitative analysis reveals that Ni is the dominant element, with an atomic percentage of 77.48% and a weight percentage of 72.73%, indicating a high concentration of NiO within the nanocomposite. Zinc is present at an atomic percentage of 12.56% and a weight percentage of 15.69%, suggesting the presence of ZnO as a secondary phase. Oxygen is also detected with an atomic percentage of 9.96% and a weight percentage of 11.58%, consistent with the metal oxide nature of the material. Minor signals for silicon (Si) and aluminum (Al) are likely due to the sample holder or substrate used during EDX analysis. The EDX results validate the successful incorporation of Zn and Ni elements in the composite, supporting the formation of a ZnO@NiO heterostructure suitable for advanced functional applications [25, 27].

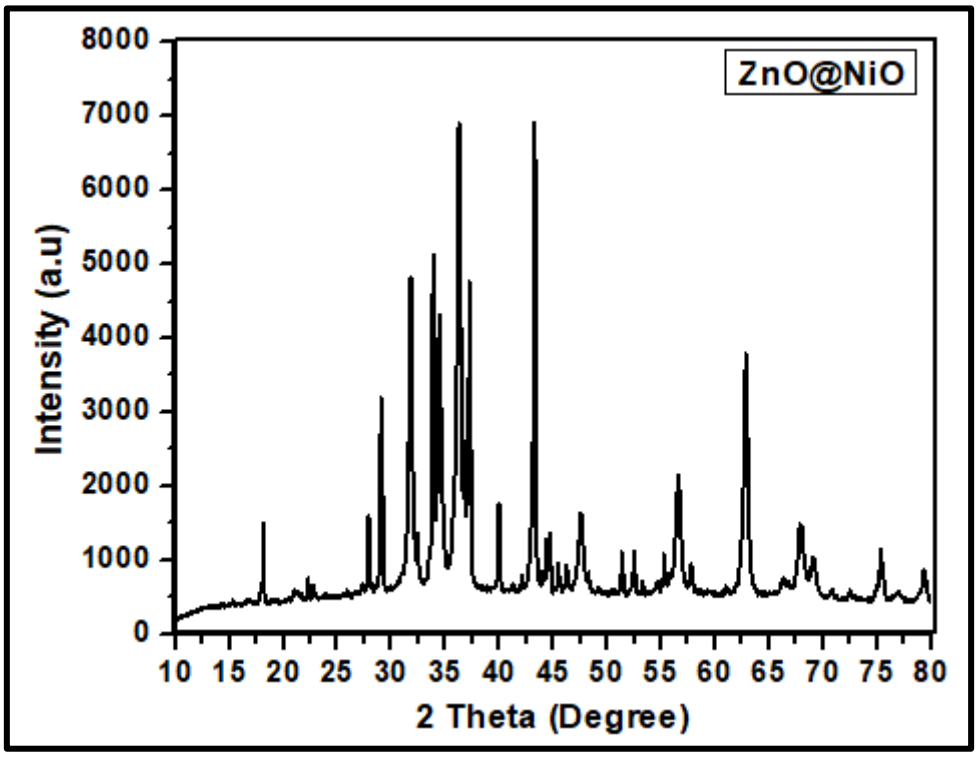

Fig. 3. XRD pattern of ZnO@NiO nanocomposite

Figure 3 displays the X-ray diffraction (XRD) pattern of the synthesized ZnO@NiO nanocomposite, illustrating the crystalline phases and structural integrity of the material. The sharp and intense diffraction peaks in the 2θ range of 10° to 80° confirm the high crystallinity of the composite [27, 28]. The peaks corresponding to ZnO are well-matched with the hexagonal wurtzite structure (JCPDS Card No. 36-1451), showing characteristic reflections at 2θ ≈ 31.77° (100), 34.42° (002), 36.25° (101), 47.54° (102), 56.60° (110), 62.86° (103), and 68.00° (112). Meanwhile, the NiO phase is identified with its cubic crystal structure (JCPDS Card No. 47-1049), with prominent diffraction peaks observed at 2θ ≈ 37.26° (111), 43.29° (200), 62.88° (220), 75.42° (311), and 79.41° (222) [28, 29]. The coexistence of both ZnO and NiO peaks without any additional impurity signals confirms the successful formation of the ZnO@NiO nanocomposite. The well-defined peaks and absence of peak broadening indicate good crystallinity and phase purity [29, 30]. The integration of ZnO and NiO at the structural level can enhance the interfacial interactions, which is beneficial for various functional applications, including photocatalysis, sensors, and optoelectronic devices.

Conclusions:

The successful synthesis of the ZnO@NiO nanocomposite using the co-precipitation method. The characterization techniques, including SEM, EDX, and XRD, confirmed the high crystallinity and phase purity of the nanocomposite, with NiO being the dominant phase, as supported by the elemental composition and crystallographic data. The strong interfacial interaction between the ZnO and NiO phases suggests that this nanocomposite holds significant potential for applications in gas sensing, catalysis, and electronic devices. The findings underscore the effectiveness of the co-precipitation method in fabricating metal oxide composites with tailored structural properties, which can be further optimized for specific technological applications. Results demonstrated the formation of a highly porous,

well-interfaced composite with distinct rod-like and plate-like crystallites. Future work should focus on exploring the performance of the ZnO@NiO nanocomposite in real-world applications, such as gas sensing and catalytic reactions, to fully understand the synergistic effects of the two phases and their potential for enhanced functionality.

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