The Bioscan AN INTERNATIONAL QUARTERLY JOURNAL OF LIFE SCIENCES 20(2): S2: 621-625, 2025 www.thebioscan.com

FABRICATION AND ANALYSIS OF NICKEL OXIDE NANOPARTICLES FOR ADVANCED APPLICATIONS

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DOI: 10.63001/tbs.2025.v20.i02.S2.pp621-625

KEYWORDS
Nickel oxide, Nanoparticles, Microwave irradiation, Electron microscopy, Magnetic properties.

Received on:

08-04-2025

Accepted on:

05-05-2025

Published on:

06-06-2025

ABSTRACT

Nickel oxide (NiO) nanoparticles were successfully synthesized using a novel microwave irradiation technique, employing glycine as a fuel. The synthesized NiO nanopowders underwent comprehensive characterization through powder X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), Fourier-transform infrared spectroscopy (FT-IR), high-resolution scanning electron microscopy (HR-SEM), and room-temperature vibrating sample magnetometry (VSM). XRD analysis confirmed the formation of a single-phase cubic NiO structure. HR-SEM images revealed nanoparticle-like morphology, corroborating the nanoscale nature of the synthesized material. Magnetic measurements via VSM exhibited a weak ferromagnetic behavior, with a saturation magnetization value of 20.93 emu/g, indicating the presence of ferromagnetic interactions at room temperature.

INTRODUCTION

Nickel oxide (NiO) is a versatile p-type semiconductor material with a wide range of technological applications, including catalysis, electrochromic devices, gas sensors, battery electrodes, and spintronic devices. Due to its remarkable optical, electrical, and magnetic properties, NiO has become a focal point of research in the development of next-generation functional materials. In particular, NiO nanoparticles exhibit enhanced surface area, tunable morphology, and size-dependent properties that are critical for improving the performance of nanodevices.The synthesis method significantly influences the structural, morphological, and magnetic characteristics of NiO nanoparticles. Traditional methods such as sol-gel, co-precipitation, and hydrothermal techniques, while effective, often require longer processing times and complex reaction conditions. In contrast, microwave-assisted synthesis has emerged as a rapid, energy-efficient, and environmentally benign technique that enables uniform heating and nucleation, leading to the formation of highly crystalline nanoparticles with controlled size and phase purity.

Recent studies have shown that NiO nanoparticles can exhibit room-temperature magnetic properties, such as weak ferromagnetism or superparamagnetism, due to uncompensated surface spins and finite-size effects. These magnetic characteristics are especially significant for their potential use in spintronic devices, where the manipulation of spin rather than charge offers the promise of faster and more energy-efficient data storage and processing. In the present study, NiO nanoparticles were synthesized using a microwave irradiation method, employing glycine as a fuel. The synthesized nanoparticles were comprehensively characterized by XRD, EDX, FT-IR, HR-SEM, and VSM to investigate their structural, morphological, and magnetic properties. The study aims to explore the suitability of these NiO nanoparticles for advanced magnetic and electronic applications.

2.LITERATURE REVIEW

Microwave-assisted synthesis is recognized for its energy efficiency, rapid processing, and uniform heating, leading to controlled particle size and crystallinity. Kalpanadevi and Manimekalai (2016) reported a simple microwave-assisted synthesis method for NiO nanoparticles, achieving good structural integrity and nanometric size distribution, suitable for functional applications [1]. Expanding this concept, Grzelczak et al. (2005) demonstrated the successful fabrication of Ni/NiO nanomaterials using microwave irradiation, highlighting the process's potential for producing magnetic nanocomposites with enhanced superparamagnetism [2]. The general versatility and environmental advantages of microwave synthesis were further emphasized in the work of Ortega-Villarreal et al. (2021), who detailed its role in green nanomaterial production, emphasizing reduced reaction times and lower energy consumption [3].

Modifying NiO with dopants further tunes its properties. Bhatt et al. (2020) synthesized lithium-doped NiO nanostructures via a facile microwave method, showing excellent optical absorption and enhanced electrochemical activity, making them promising candidates for energy storage devices [4].

Beyond microwave synthesis, other eco-friendly or efficient routes have been explored. Rana and Jeevanandam (2023)

synthesized NiO nanoparticles by calcining surfactant-intercalated layered hydroxides, resulting in mesoporous particles with high surface areas and significant adsorption capacity for environmental contaminants [5]. Magnetic properties of NiO nanoparticles are of great scientific interest. Duan et al. (2015) investigated size-dependent magnetic behavior, focusing on the blocking temperature and Néel temperature, providing insights into finite-size effects in antiferromagnetic systems [6]. Similarly, Moumen et al. (2019) prepared Ni colloidal nanoparticles and explored their magnetic response, showing a clear size and morphology correlation [7]. Roy et al. (2018) demonstrated that carbon-coated Ni/NiO core-shell particles exhibited large magnetoresistance, suggesting a strong interplay between interface structure and magnetic behavior [8].

The synthesis method significantly impacts particle properties. Romanova and Kirillov (2018) used a citric acid-aided route to synthesize NiO along with Cu and Co oxides, achieving uniform morphology and high thermal stability [9]. Pulimi and Jeevanandam (2009) investigated the effect of anions during homogeneous precipitation of NiO and their influence on magnetic behavior, revealing that anion type modulates surface structure and hence the magnetic properties [10]. The exploration of various synthesis techniques for NiO nanoparticles continues to reveal significant advancements in tailoring their physical and functional properties. In particular, hydrothermal synthesis offers precise control over morphology and porosity. Nguyen et al. (2018) compared nanoporous NiO nanowires and nanosheets synthesized hydrothermally, demonstrating that nanowire structures exhibit superior electrochemical properties due to enhanced charge transport pathways and high surface area [11].

Wet chemical routes, including organo-templated synthesis, also provide access to well-defined nanostructures. Zahra and Ahmad (2020) synthesized cubic-shaped NiO nanoparticles using an organo-template wet approach. Their work confirmed phase purity, optical band gap tunability, and promising electrochemical behavior for capacitor applications [12, 19]. Similarly, Li et al. (2018) conducted an in situ structural investigation of Ni/NiOx supercapacitor electrodes during cyclic operation. Their polarized neutron and X-ray reflectometry studies revealed dynamic interface changes during charge-discharge cycles, highlighting structural stability as key to device longevity [13].

Though not directly about NiO, the work of Yu et al. (2009) on MnO_2 nanostructures synthesized via a microwave-assisted emulsion method offers insights into pseudocapacitance behavior and synthetic strategies applicable to NiO systems [14].

Biotemplating offers another novel route to nanostructured materials. Rani et al. (2010) utilized fibrinogen lyophilisomes as bio-templates to synthesize nickel oxide and hydroxide nanoparticles, achieving uniform morphology and biocompatibility, indicating potential for biomedical applications [15].

The biological and environmental applications of NiO are gaining traction. Anand et al. (2020) explored the antimicrobial efficacy of NiO nanoparticles, confirming that size and surface morphology significantly influence bactericidal activity, alongside structural and optical properties [16]. Jawad and Hassan (2023) further advanced this by performing a green synthesis of NiO nanoparticles incorporating carbon dots, enhancing both antimicrobial performance and eco-friendliness of the process [17].

Studies by Kumar and Prasad (2011) focused on identifying biomolecules in *Tephrosia tinctoria*, which can potentially act as reducing and stabilizing agents in green synthesis of metal oxide nanoparticles [18, 20].

Finally, Singh et al. (2023) reported on 3D microporous nickel/nickel oxide nanoflakes with enhanced electrochemical performance for supercapacitor applications. The hierarchical porosity and Ni/NiO interface synergistically contributed to high capacitance and long cycle stability, making them excellent candidates for next-generation energy storage [21].

3. EXPERIMENTAL

3.1 PREPARATION OF NIO NANOPARTICLES BY MICROWAVE IRRADIATION METHOD

In this synthesis method, nickel nitrate hexahydrate ($Ni(NO_3)_3 \cdot 6H_2O$) acts as the oxidizing agent, while glycine serves both as a reducing agent and a fuel. Stoichiometric amounts of nickel nitrate and glycine were each dissolved in 20 mL of distilled water. The solutions were mixed in an alumina crucible and stirred thoroughly for 15 minutes. The mixture was then subjected to microwave irradiation for 10 minutes. During this process, the solution rapidly vaporized and transformed into a solid mass. The resulting solid was washed with ethanol to remove impurities and subsequently dried in an electric oven at 80 °C for 2 hours. The final product was designated as NiO.

3.2. CHARACTERIZATIONS

Structural analysis was carried out using a Philips X'pert X-ray diffractometer with Cu-Kα radiation (λ = 1.540 Å). The presence of functional groups was examined using Fourier-transform infrared (FT-IR) spectroscopy (PerkinElmer). Morphological features and elemental composition were analyzed using high-resolution scanning electron microscopy (HR-SEM, JEOL JSM-6360). Magnetic properties, including saturation magnetization (Ms), remanent magnetization (Mr), and coercivity (Hc), were investigated at room temperature using a vibrating sample magnetometer (VSM).

4. RESULTS AND DISCUSSION

4.1. POWDER XRD ANALYSIS

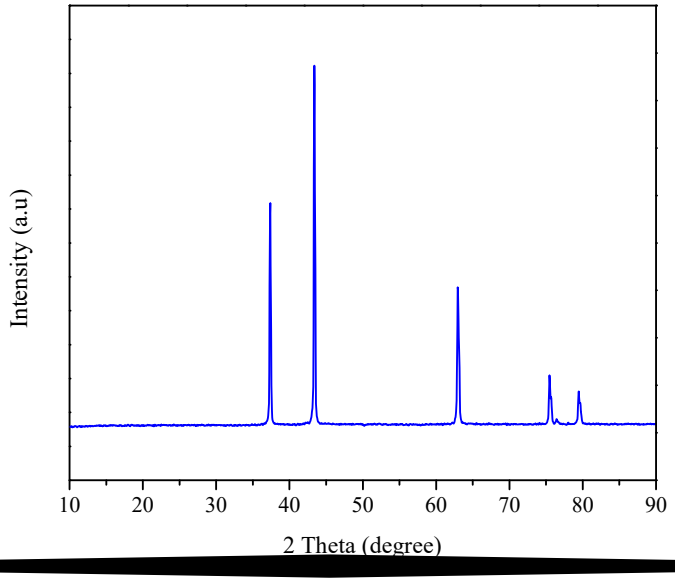

Figure 1. Powder XRD result of NiO sample.

The average crystallite size, structural and phase purity of NiO nanoparticles was analyzed by powder XRD pattern. Fig. 1 shows the powder XRD pattern of NiO sample. The XRD diffraction peaks at 2 θ values positioned at 37.35, 43.38, 62.93, 75.47, and 79.46°, matching to (111), (200), (220), (311) and (222) planes (JCPDS No. 78-0643), showed the formation of a single phase and pure cubic NiO, which confirmed that the microwave heating completely decomposed the precursors and produced NiO nanoparticles. The average crystallite size of NiO nanoparticles was calculated using Debye Scherrer's formula given in Eq. (1)

$$L = \frac{0.89\lambda}{\beta \cos\theta}$$

---- (1)

where L is the crystallite size, θ, the Bragg diffraction angle, λ is the X-ray wavelength, and β is the full width at half maximum (FWHM). The average crystallite size is 17.15 nm.

The lattice parameter of NiO nanoparticles was calculated using the formula given in Eq. (2):

$$\sin^2\theta = \frac{\lambda^2}{4}\left[\frac{4}{3}\left(\frac{h^2+hk+k^2}{a^2}\right)+\frac{l^2}{c^2}\right]$$

---- (2)

where θ is the diffraction angle, h, k, and l are Miller's indices and λ is the incident wavelength (λ = 1.540 Å). The lattice parameter of NiO sample is found to be a = 5.012 Å and c= 13.695 Å, which are extremely close to the reported values (a = 5.033 Å and c = 13.753 Å, JCPDS No. 89-0598).

4.2. FT-IR SPECTROSCOPY

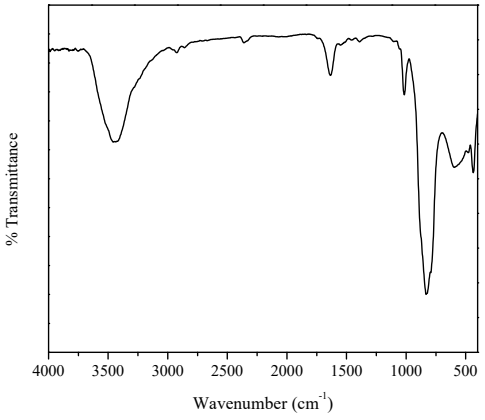

Figure 2. FT-IR spectra of NiO sample

Figure 2 presents the FT-IR spectrum of the NiO sample. A broad absorption band observed in the range of ~3450 to 3250 cm^{-1} corresponds to the O–H stretching vibrations of surface-adsorbed water molecules, indicating the presence of a significant amount of surface hydroxyl groups. The band around ~1610 cm^{-1} is attributed to the bending vibrations of H–O–H from molecular water. Additionally, a strong absorption band in the range of 850–450 cm^{-1} is characteristic of Ni–O stretching vibrations, confirming the formation of NiO.

4.3. SEM STUDIES

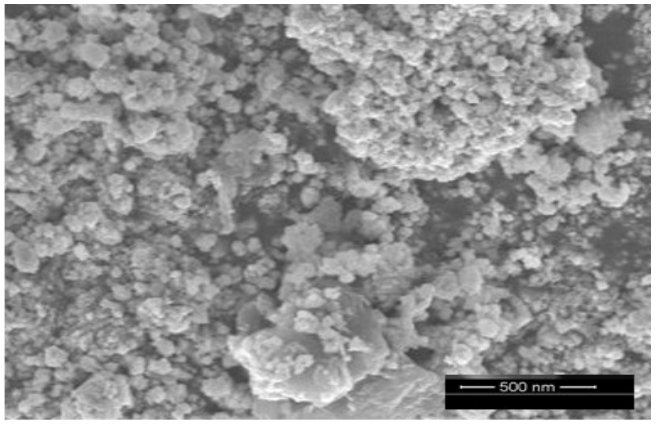

Figure 3. HR-SEM image of NiO sample.

The surface morphology of the NiO powder was examined using high-resolution scanning electron microscopy (HR-SEM). Figure 3 displays the HR-SEM image of the NiO nanostructure. The image reveals a particle-like morphology with evidence of smaller agglomerated nanocrystals. This suggests that the formation of NiO nanocrystals occurred during the microwave irradiation process, leading to the observed agglomeration and nanoscale features.

4.4 EDX ANALYSIS

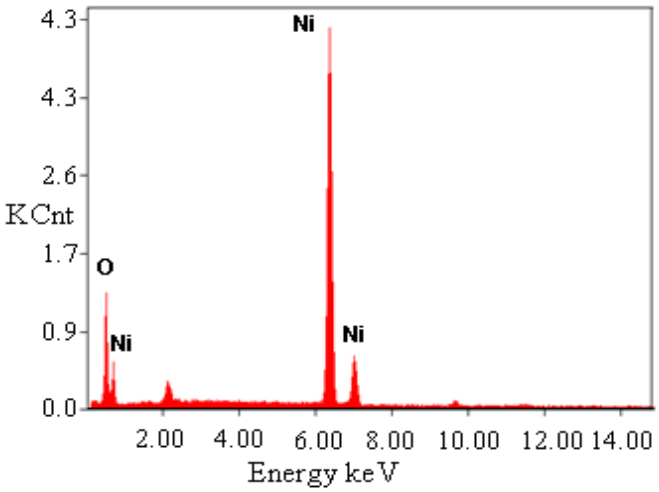

Figure 4. EDX spectrum of NiO sample.

The elemental analysis and purity of NiO nano-crystals was determined by EDX spectra as shown in Fig. 4. The EDX analysis showed the presence of Ni and O elements by the manifestation of Ni and O peaks with no any other impurity peaks. Hence, the microwave irradiation method reactions are authoritative evidence to propose that the sample NiO product only.

4.5 VSM MEASUREMENTS

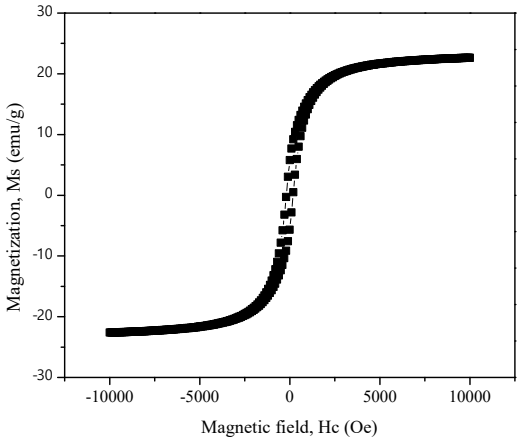

Figure 5. VSM hysterisis loop of NiO sample.

The magnetic properties of the NiO nanoparticles, including saturation magnetization (Ms), remanent magnetization (Mr), and coercivity (Hc), were analyzed using vibrating sample magnetometry (VSM). Figure 5 presents the room-temperature VSM hysteresis loop for NiO nanoparticles measured in a magnetic field range of –10,000 to +10,000 Oe. Nickel oxide (NiO), a well-known magnetic semiconductor metal oxide, has garnered attention due to its potential applications in various interdisciplinary fields.

The analysis revealed relatively low values of coercivity (Hc), saturation magnetization (Ms), and remanent magnetization (Mr), indicating soft ferromagnetic behavior. The VSM results confirm that the NiO nanoparticles synthesized by the microwave irradiation method exhibit notable ferromagnetic properties. Specifically, the saturation magnetization (Ms) was measured to be 20.93 emu/g, the remanent magnetization (Mr) was 0.901 emu/g, and the coercivity (Hc) was 14.43 Oe. These findings highlight the effectiveness of the microwave irradiation technique in producing NiO nanoparticles with significant ferromagnetic behavior.

CONCLUSION

NiO nanoparticles were synthesized using a simple and rapid microwave irradiation method, employing nickel nitrate as the precursor and glycine as the fuel. X-ray diffraction (XRD) analysis confirmed the formation of a single-phase, pure NiO structure, with crystallite sizes ranging from approximately 15 to 20 nm. High-resolution scanning electron microscopy (HR-SEM) revealed a nanoparticle-like morphology. Vibrating sample magnetometry (VSM) analysis indicated that the NiO nanoparticles exhibited ferromagnetic behavior, with a high saturation magnetization value of 20.93 emu/g.

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