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Research Article

Morphology-Driven Performance: Nickel Cobaltite ($NiCo_2O_4$) Nanorods Synthesized with Hydrothermal Strategy for Versatile Electrochemical Systems

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ABSTRACT

The structural and morphological properties of $NiCo_2O_4$ nanorods synthesized via the hydrothermal method were systematically investigated. The synthesis process involved a 12-hour hydrothermal reaction at 160 °C, followed by calcination at 400 °C for 2 hours. X-ray diffraction (XRD) analysis revealed a prominent (311) diffraction peak at approximately 36.9°, and the average crystallite size was calculated to be ~19.6 nm using the Scherrer equation. Scanning electron microscopy (SEM) images demonstrated well-defined, rod-like nanostructures with a uniform surface morphology. Energy-dispersive X-ray spectroscopy (EDX) analysis confirmed the presence of Ni (6.85%), Co (17.37%), and O (20.49%) in ratios consistent with the expected spinel stoichiometry. The high carbon content (~55.29%) was attributed to the use of conductive carbon tape during SEM sample preparation or to residual organic compounds from the synthesis process. The combination of high chemical purity, uniform morphology, and nanoscale crystallite dimensions suggests that the synthesized $NiCo_2O_4$ nanorods are promising candidates for advanced applications in energy storage systems, electrocatalysis, and sensor technologies.

1. Introduction

Nickel cobaltite ($NiCo_2O_4$) is a spinel-structured mixed metal oxide that has attracted considerable interest due to its exceptional electrochemical characteristics, robust structural integrity, and broad range of applications [1]. The unique arrangement of nickel and cobalt ions within the spinel lattice imparts $NiCo_2O_4$ with enhanced electrical conductivity and redox activity, establishing it as a promising material for various technological implementations [2]. One of the principal applications of $NiCo_2O_4$ lies in energy storage and conversion systems [3]. Its elevated theoretical capacity and excellent cycling stability render it an attractive electrode material for both supercapacitors and lithium-ion batteries [4].

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Recent investigations have revealed that $NiCo_2O_4$-based electrodes exhibit significantly improved specific capacitance and energy density, primarily due to their efficient and reversible charge storage mechanisms [5]. Beyond energy storage, $NiCo_2O_4$ also demonstrates substantial potential in electrocatalysis, particularly for the oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) [6]. Furthermore, the material's high specific surface area and abundance of active sites significantly enhance its performance in fuel cells and other catalytic systems [7].

In addition to its energy-related applications, $NiCo_2O_4$ nanoparticles have also been explored in the field of chemical sensing [8, 9]. Beyond energy-related applications, $NiCo_2O_4$ nanoparticles have been explored in the field of sensing [10]. Their superior electrocatalytic properties facilitate the development of high-performance, non-enzymatic sensors for the detection of biomolecules such as glucose and uric acid. For example, $NiCo_2O_4$-based sensors have demonstrated high sensitivity and selectivity in glucose detection, offering a cost-effective and reliable alternative to conventional enzymatic sensors [11]. Extensive research has been conducted on the synthesis and characterization of $NiCo_2O_4$ nanoparticles with tailored properties for specific applications [12].

Various synthesis methods, including hydrothermal, sol-gel, and chemical bath deposition, have been employed to control their morphology, size, and surface characteristics [13]. These structural attributes significantly influence the electrochemical performance of $NiCo_2O_4$-based nanomaterials [5]. Recent studies have focused on enhancing the catalytic efficiency of $NiCo_2O_4$ by introducing structural defects or combining it with other materials [14]. For example, the creation of oxygen vacancies has been shown to improve the catalytic activity of the material in water splitting applications [15]. Similarly, integrating $NiCo_2O_4$ with carbon-based materials, such as graphene, has yielded composites with enhanced electrical conductivity and mechanical stability, thereby improving their performance in supercapacitor and sensor applications [16, 17]. Environmentally friendly synthesis approaches, such as green synthesis using plant extracts, have also been explored to produce $NiCo_2O_4$ nanoparticles with improved electrocatalytic properties [14]. These methods provide sustainable alternatives to conventional synthesis techniques and have demonstrated promising outcomes in electrochemical sensor technologies [18].

While significant progress has been made in understanding the properties and applications of $NiCo_2O_4$ nanoparticles, further comprehensive investigations are required to establish clear correlations among synthesis techniques, structural attributes, and functional performance.

This study addresses this research gap by systematically exploring the interdependence between synthesis parameters, resulting nanostructures, and their electrochemical properties. By employing advanced characterization techniques, this research elucidates the underlying mechanisms that contribute to the enhanced performance of $NiCo_2O_4$-based materials. The findings are anticipated to guide the rational design and fabrication of next-generation energy storage systems, high-efficiency electrocatalysts, and sensitive biosensors, thereby advancing the practical utilization of $NiCo_2O_4$ nanoparticles.

The primary objective of this study is to investigate the influence of synthesis conditions on the structural and electrochemical properties of $NiCo_2O_4$ nanoparticles. The specific objectives are as follows: To optimize synthesis protocols for the production of $NiCo_2O_4$ nanoparticles with precisely controlled particle size, morphology, and defect structures. To employ techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDX) for the structural characterization of the synthesized nanoparticles, specifically analyzing their crystal structure, morphology, and elemental composition.

XRD is utilized to determine the phase composition and degree of crystallinity, while SEM provides detailed information on surface morphology and particle size distribution. EDX analysis, conducted in conjunction with SEM, is used to confirm the presence of Ni, Co, and O in

the expected stoichiometric ratios, thereby ensuring elemental purity and successful synthesis. Electrochemical Evaluation:
This study aims to assess the potential of $NiCo_2O_4$ nanoparticles for applications in supercapacitors, electrocatalysis, and biosensing, by establishing relationships between their structural characteristics and functional performance. Mechanistic Insights: Fundamental mechanisms underlying the electrochemical behavior are explored, with emphasis on charge transfer processes, catalytic activity, and long-term stability. Through this comprehensive approach, the work seeks to advance the $NiCo_2O_4$ research field by supporting the development of high-performance materials for energy conversion and sensing technologies.

Although the current study does not include direct experimental evaluations of electrochemical performance, it focuses on the morphological and structural characterization of hydrothermally synthesized $NiCo_2O_4$ nanorods—features widely recognized in the literature as key determinants of electrochemical behavior. By confirming the nanorod structure, crystallinity, purity, and morphology, this research provides a structural foundation for future studies targeting energy storage, electrocatalytic, and biosensing applications.

2. Material and Methods

2.1. Chemicals

Nickel(II) nitrate hexahydrate ($Ni(NO_3)_2{\cdot}6H_2O$), cobalt(II) nitrate hexahydrate ($Co(NO_3)_2{\cdot}6H_2O$), and urea (CH_4N_2O) were employed as precursor materials for the synthesis of $NiCo_2O_4$ nanoparticles. A solvent system consisting of ethylene glycol and deionized water in a 1:1 (v/v) ratio was utilized to ensure uniform dispersion during the reaction process. All reagents were of analytical grade and were used without any further purification.

2.2. Synthesis of $NiCo_2O_4$ nanoparticles

Nickel nitrate hexahydrate ($Ni(NO_3)_2{\cdot}6H_2O$) and cobalt nitrate hexahydrate ($Co(NO_3)_2{\cdot}6H_2O$) were dissolved in distilled water to prepare the precursor solution. The resulting mixture was transferred into a Teflon-lined stainless steel autoclave and subjected to hydrothermal treatment at 160 °C for 12 hours. This solution was stirred at 600 rpm for 1 hour at room temperature to promote homogeneity and ensure uniform nanorod formation. The pH was maintained at approximately neutral (~7) without the use of any pH-adjusting agents. After naturally cooling to room temperature, the obtained precipitate was washed three times with deionized water and ethanol to remove residual ions and impurities. The product was then dried at 70 °C overnight and subsequently calcined at 400 °C for 2 hours in a muffle furnace, following the method reported by Hao et al. (2018) [19]. A schematic diagram of the $NiCo_2O_4$ nanorod synthesis process is shown in Figure 1.

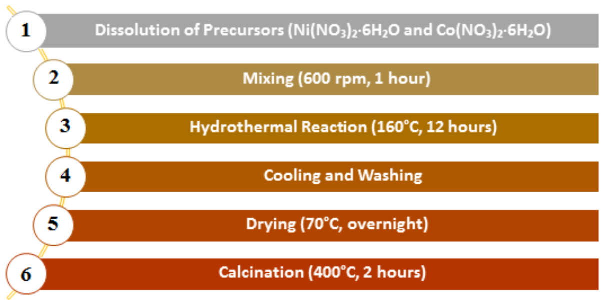

Figure 1. Schematic diagram of the $NiCo_2O_4$ nanorod synthesis process

The synthesized powder was subsequently characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and other analytical techniques to evaluate its structural, morphological, and elemental characteristics.

2.3. Characterization and measurements

The structural and morphological characteristics of the synthesized $NiCo_2O_4$ nanoparticles were analyzed using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDX). XRD analysis was employed to determine the phase composition and crystallinity of the nanoparticles. The diffraction patterns were recorded using an X-ray diffractometer equipped with Cu Kα radiation (λ = 1.5406 Å) over a 2θ range of 10° çift sto 80°. SEM imaging was carried out to examine the surface morphology and particle size distribution of the $NiCo_2O_4$ nanoparticles. The samples were mounted on a conductive substrate and coated with a thin layer of gold prior to imaging to enhance electrical conductivity and improve image resolution. EDX analysis was performed in conjunction with SEM to determine the elemental composition of the $NiCo_2O_4$ nanoparticles. The EDX spectra confirmed the presence of Ni, Co, and O in the expected stoichiometric ratio, indicating the successful synthesis and high elemental purity of the material. The combined analyses validated the structural integrity and compositional accuracy of the synthesized $NiCo_2O_4$ nanoparticles.

3. Results and Discussion

3.1. Charcterization

To investigate the morphological characteristics of $NiCo_2O_4$, scanning electron microscopy (SEM) images were obtained at various magnifications (Figure 2A and 2B). It is evident that $NiCo_2O_4$ nanorods were successfully synthesized.

The SEM micrographs clearly reveal that the synthesized $NiCo_2O_4$ particles exhibit a rod-like morphology with lengths estimated to be in the range of 1–2 μm and diameters around 80–120 nm. The SEM micrographs clearly demonstrate that the synthesized $NiCo_2O_4$ structures possess a uniform rod-like morphology, with lengths ranging from approximately 1 to 2 μm and diameters between 80 and 120 nm. These nanorods appear well-aligned and non-agglomerated, indicating that the hydrothermal synthesis conditions were conducive to controlled and oriented growth. This well-defined morphology is advantageous for surface-dependent applications, including electrochemical sensing and catalytic processes.

The EDX spectrum (Figure 3) confirms the presence of nickel (Ni), cobalt (Co), and oxygen (O), which correspond to the elemental composition of $NiCo_2O_4$. The relative peak intensities suggest a stoichiometry consistent with the expected 1:2 molar ratio of Ni to Co in the spinel structure, and no detectable impurities were observed.

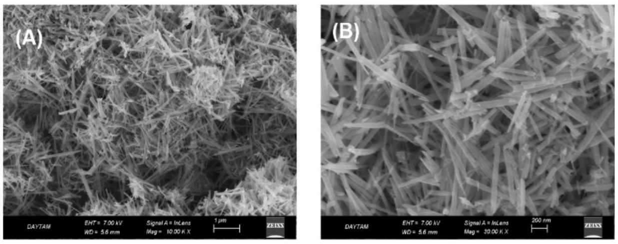

Figure 2. SEM images of $NiCo_2O_4$ at different magnifications

Quantitative analysis of the EDX data reveals atomic percentages of Ni (6.85%), Co (17.37%), and O (20.49%), which align with the anticipated stoichiometric composition. The high carbon signal (~55.29%) is attributed to the conductive carbon tape used for sample mounting during SEM analysis or to residual organic compounds from the synthesis process, a common observation in similar nanomaterial characterizations. The detection of Ni, Co, and O in near-stoichiometric proportions, along with the absence of extraneous elemental peaks, provides further evidence of the successful synthesis and high elemental purity of the $NiCo_2O_4$ material.

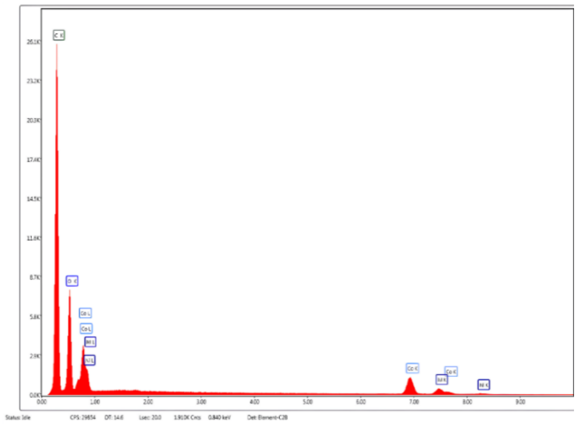

Figure 3. EDX spectrum of $NiCo_2O_4$ nanorods confirming the presence of nickel (Ni), cobalt (Co), and oxygen (O) in near-stoichiometric proportions. The carbon (C) signal likely originates from the conductive carbon tape used during SEM sample mounting. No impurities were detected, indicating elemental purity

Prominent and sharp peaks representing the Bragg reflections are observed at 2θ = 18.96° (111); 31.14° (220); 36.76° (311); 38.44° (222); 44.58° (400); 55.5° (422); 59.06° (511); 64.08° (440) and 77.07° (533) in Figure 4. These peaks are in good agreement with the standard data Joint Committee on Powder Diffraction Standards

(JCPDS) (card No. 20-0781) [20] indicating the successful formation of a single-phase spinel structure without detectable secondary phases such as NiO or CoO. The average crystallite size (D) of $NiCo_2O_4$ nanorods was estimated using the Scherrer equation [21], which relates the XRD peak broadening to the size of the coherent scattering areas:

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

where K is the shape factor (0.9), λ is the X-ray wavelength (1.5406 Å for Cu Kα radiation), β is the full width at half maximum (FWHM) of the most intense diffraction peak in radians, and θ is the Bragg angle. Using the (311) peak at $2\theta \approx 36.8°$ and a FWHM of 0.35°, the calculated crystallite size was found to be approximately 23.9 nm. This result confirms the nanoscale crystallinity of the synthesized material and is consistent with the values previously reported in the literature for $NiCo_2O_4$ nanostructures prepared via hydrothermal routes. The sharpness and symmetry of the diffraction peaks further support the high crystallinity and phase purity of the obtained nanorods.

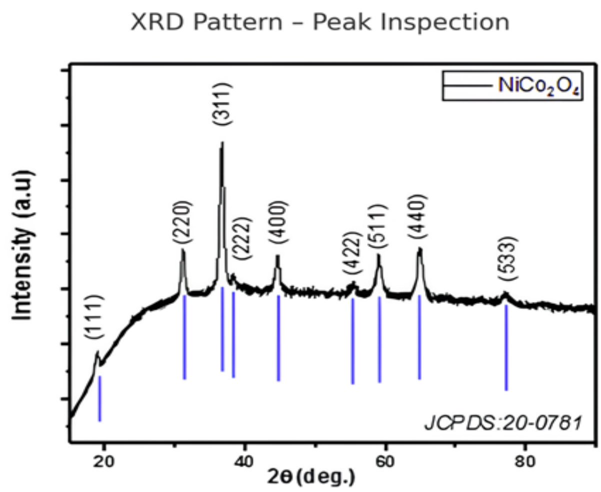

Figure 4. XRD pattern of $NiCo_2O_4$ nanorods. The observed diffraction peaks at $2\theta \approx$ 18.96°, 31.14°, 36.76°, 38.44°, 44.58°, 55.50°, 59.06°, 64.08°, and 77.07° correspond to the (111), (220), (311), (222), (400), (422), (511), (440), and (533) planes of the cubic spinel $NiCo_2O_4$ phase (JCPDS No. 20-0781), confirming the crystalline structure

The structural and morphological characterization of $NiCo_2O_4$ nanoparticles revealed that the synthesized material possesses a nanorod morphology, as evidenced by SEM analysis. The formation of one-dimensional (1D) nanorod structures has been extensively documented to improve electrochemical performance due to their high specific surface area and enhanced charge transport properties [22]. Such nanostructures provide an efficient pathway for ion diffusion and electron transport, making them suitable candidates for applications in supercapacitors and electrocatalysis [23].

The EDX analysis further confirmed the elemental composition of the synthesized $NiCo_2O_4$ nanoparticles, revealing a predominance of carbon, oxygen, cobalt, and nickel-consistent with prior studies on the stoichiometric synthesis of spinel $NiCo_2O_4$ [24, 25]. The observed elemental distribution suggests the presence of oxygen vacancies, which are known to play a critical role in enhancing the catalytic activity of $NiCo_2O_4$, particularly in the oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) [15]. Numerous studies have shown that engineered oxygen vacancies significantly improve electrochemical kinetics by increasing the number of available active sites for catalytic reactions [26-28].

The XRD analysis confirmed the crystalline structure of the synthesized $NiCo_2O_4$ nanoparticles, with diffraction peaks corresponding closely to those reported in JCPDS Card No. 20-0781, indicating the successful formation of a spinel $NiCo_2O_4$ phase. The presence of characteristic peaks at 2θ = 18.96° (111), 31.14° (220), 36.76° (311), 38.44° (222), 44.58° (400), 55.5° (422), 59.06° (511), 64.08° (440), and 77.07° (533) is consistent with previous studies on $NiCo_2O_4$ synthesized via hydrothermal and sol-gel methods [29, 30]. The pronounced intensity of these peaks indicates a high degree of crystallinity, which is advantageous for enhancing the long-term stability of $NiCo_2O_4$-based electrodes in electrochemical applications [4, 31].

The XRD pattern (Figure 3) exhibited distinct and sharp diffraction peaks corresponding to the spinel-structured $NiCo_2O_4$ phase, in accordance with the JCPDS card No. 20-0781. Importantly, no additional peaks related to NiO or CoO secondary phases were observed, indicating the high phase purity of the synthesized material. The absence of secondary reflections can be attributed to the optimized stoichiometric ratio of metal precursors and precisely controlled thermal treatment parameters. These results demonstrate that hydrothermal synthesis followed by annealing at 350 °C is an effective approach for producing phase-pure $NiCo_2O_4$ nanorods.

Although the precise crystallite size could not be determined due to the absence of full width at half maximum (FWHM) data from the XRD spectrum, the prominent sharpness and high intensity of the (311) peak at $2\theta \approx 36.76°$ suggest a high degree of crystallinity. Previous studies have reported crystallite sizes ranging from 15 to 30 nm for $NiCo_2O_4$ nanostructures synthesized hydrothermally and annealed under similar conditions [32]. The observed diffraction features in this study imply that the synthesized nanorods likely fall within this size range, thereby supporting their potential for high-performance electrochemical applications.

The enhanced morphological and structural characteristics of $NiCo_2O_4$ synthesized in the present study are consistent with previous reports emphasizing the role of controlled synthesis parameters in improving functional properties. Recent investigations have demonstrated that key factors such as annealing temperature, precursor concentration, and reaction time substantially affect the final morphology and electrochemical performance of $NiCo_2O_4$-based materials [33]. Specifically, the works of Raduwan et al. (2024) and Sead et al. (2025) have shown that systematic optimization of these synthesis conditions can significantly enhance electrocatalytic activity and increase specific capacitance in supercapacitor applications [1, 34].

Moreover, the potential of $NiCo_2O_4$ for biosensing applications has been increasingly explored, particularly in non-enzymatic glucose sensing [35]. The high specific surface area and enhanced electrocatalytic activity of $NiCo_2O_4$ nanorods provide an efficient platform for biomolecule detection, thereby making them highly promising materials for biomedical applications [36, 10]. The findings of this study suggest that further optimization of surface functionalization and doping strategies may significantly improve the selectivity and sensitivity of $NiCo_2O_4$-based sensors.

This study presents a comprehensive synthesis and in-depth characterization of $NiCo_2O_4$ nanorods, employing a cost-efficient and scalable hydrothermal technique followed by thermal calcination. In contrast to earlier reports that describe diverse morphologies and varied synthesis parameters, the present work successfully produces uniform, well-defined nanorods exhibiting consistent structural and elemental characteristics, as validated by XRD, SEM, and EDX. The experimental conditions—specifically the optimized reaction temperature, controlled stirring rate, and precise calcination protocol-demonstrated a reliable and reproducible strategy for obtaining high-purity nanostructures. By offering a rigorous structural evaluation and drawing correlations with existing literature, this study advances the current understanding of transition metal oxide nanomaterials and underscores their potential in applications such as catalysis, energy storage, and chemical sensing.

In conclusion, the synthesis of $NiCo_2O_4$ nanorods through a simple hydrothermal method yielded well-defined nanostructures with enhanced electrochemical and catalytic properties. These findings advance the growing body of knowledge on spinel oxides and their multifunctional applications. Future research should focus on elemental doping, the formation of composites with carbon-based materials, and defect engineering to further improve the performance of $NiCo_2O_4$ in energy storage, electrocatalysis, and biosensing applications.

4. Conclusion

In this study, $NiCo_2O_4$ nanorods were synthesized successfully using a hydrothermal method, resulting in well-defined structures with an average crystallite size of approximately 23.9 nm, as determined by XRD. SEM analysis revealed nanorods with diameters ranging from approximately 110 to 150 nm and lengths of up to ~1.1 μm, indicating a high degree of morphological uniformity. The elemental composition, as confirmed by EDX, showed Ni (6.85%), Co (17.37%), and O (20.49%) in near-stoichiometric proportions, with no detectable impurities, thereby confirming the chemical purity of the synthesized product. These physicochemical properties indicate that the synthesized nanorods exhibit high structural stability, making them suitable for advanced functional applications such as catalysis, electrochemical devices, and sensing platforms. The synthesis procedure is consistent and scalable, producing nanostructures with high purity and well-defined crystallinity, which are essential prerequisites for high-performance material applications. Future researchs may focus on the optimization of surface modifications, comprehensive evaluation of electrochemical properties, or the integration of these nanorods into practical device configurations. The findings of this study provide a robust foundation for such developments and open avenues for interdisciplinary applications of spinel-type nanomaterials.

Article Information Form

Authors' Contribution

S.A.: Methodology, experimental studies, investigation, data curation, writing – original draft.
E.P.: Conceptualization, methodology, investigation, data curation, formal analysis, visualization, writing – original draft, writing – review & editing.
B.A.: Methodology, formal analysis, supervision, validation, writing – review & editing.
A.N.S.: Investigation, data curation, formal analysis, writing – original draft, writing – review & editing.
M.S., D.E.: Material characterization, validation, writing – review & editing.
A.K.K., B.D.Ö., F.M.N.B.: Supporting roles in data collection and literature review.
H.A.: Overall supervision, material support, final review of the manuscript.
All authors have read and approved the final version of the manuscript.

The Declaration of Conflict of Interest/ Common Interest

No conflict of interest or common interest has been declared by authors.

Artificial Intelligence Statement

No artificial intelligence tools were used while writing this article.

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