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Research Article

Synthesis, Structural, And Electrochemical Studies Of Nanostructured $Niwo_4$ As Anode For Sodium-Ion And Lithium-Ion Batteries

Mamatharani. V[1*], Satish Kumar[2]

[1*]Resarch Scholar, Department of Physics, Mansarovar Global University, Bhopal, Madhya Pradesh.
[2]Resarch Guide, Department of Physics, Mansarovar Global University, Bhopal, Madhya Pradesh.

***Corresponding author:** Mamatharani. V
*E-mail: mamatharani.159@gmail.com

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ABSTRACT

In this work used a chemical precipitation approach to create nanostructured nickel tungstate ($NiWO_4$), which was then heated to temperatures between 600 and 700 °C. The X-ray diffraction (XRD) investigation indicated that the material calcinated at 700 °C had improved phase purity and crystallinity. The electronic structure and surface chemical states were examined using X-ray photoelectron spectroscopy (XPS). This confirmed that Ni^{2+} and W^{6+} were in stable oxidation states and showed that the material was electrochemically ready for charge transfer reactions. Both the lithium-ion and sodium-ion half-cells tested electrochemically performed admirably, exhibiting long-term cycle stability, a favorable rate capability, and a high specific capacity. Thermotreatment improved structural integrity and adjusted surface chemistry for ion intercalation, which led to these enhancements. Results from studies comparing hybrid tungsten oxide systems with rGO-composited $NiWO_4$ electrodes lend credence to the idea that surface-level alterations play a part in improving performance. According to our results, thermally engineered $NiWO_4$ has great promise as a bifunctional anode material for future technologies that combine lithium-ion and sodium-ion batteries.

Keywords: Nickel tungstate; $NiWO_4$; Lithium-ion battery; Sodium-ion battery; Nanostructures; Electrochemical performance; XRD; Cyclic voltammetry; EIS

1. INTRODUCTION

The increasing global emphasis on sustainable and efficient energy storage has expedited the quest for improved, cost-effective alternatives to traditional Sodium-ion batteries (SIBs), lithium-ion batteries (LIBs) have gathered considerable attention among developing technologies owing to the plentiful and inexpensive nature of sodium, positioning them as a viable alternative in light of escalating lithium pricing and constrained availability. Enhancing the performance of both LIBs and SIBs necessitates the development of high-capacity electrode materials that exhibit superior durability and rapid charge-discharge capabilities. Transition metal tungstates have garnered interest due to their adaptable crystal forms, intrinsic chemical stability, and elevated theoretical capacities.

Nickel tungstate ($NiWO_4$), a binary transition metal oxide, has demonstrated potential as an anode material for sodium-ion batteries (SIBs) and lithium-ion batteries (LIBs). The electrochemical performance is augmented by the synergistic redox activity of nickel and tungsten, facilitating superior charge storage, greater electron conductivity, and mechanical durability (Amudha et al., 2023; Tang et al., 2024). Research conducted by Zong et al. (2022) and Saghaei & Shabani-Nooshabadi (2024) illustrates that nanoscale engineering of $NiWO_4$ enhances its active surface area, improves electrolyte accessibility, and elevates ion transport efficiency.

Furthermore, the integration of conductive carbon materials, including carbon nanotubes (CNTs) and reduced graphene oxide (rGO), markedly enhances electrical conductivity and mitigates structural degradation during cycling (Amudha et al., 2023; Pan et al., 2023). These hybrid architectures permit electrodes to provide high-

rate performance and sustained operational stability. The morphology and crystallinity of $NiWO_4$ nanostructures can be modified using many synthesis methods, such as sol-gel, hydrothermal, and co-precipitation approaches, facilitating customized characteristics for advanced battery applications.
Despite considerable advancements, researchers still lack a thorough understanding of how synthesis techniques, structural characteristics, and electrochemical performance correlate—especially in terms of the dual compatibility of $NiWO_4$ with sodium-ion battery (SIB) and lithium-ion battery (LIB). This study seeks to examine the synthesis routes systematically, structural properties, and electrochemical performance of nanostructured $NiWO_4$, emphasizing the correlation between morphological and compositional factors and battery performance metrics to evaluate its potential for next-generation energy storage applications.

2.LITERATURE REVIEW

Samriddhi Saxena (2025) conducted a comprehensive assessment of Mn-Fe-Ni layered oxide materials intended for sodium-ion battery cathodes. The investigation indicated that optimizing the ratios of transition metals and regulating phase distribution can significantly enhance electrochemical performance. The utilization of sophisticated characterization instruments provided a more lucid comprehension of material dynamics. The results highlight the appropriateness of Mn-Fe-Ni oxides as cost-effective, high-performance cathodes for sodium-ion storage.
Yunlei Wang (2024) provided a comprehensive analysis of silicon-based anodes for lithium-ion batteries, emphasizing the synthesis and structural enhancement of silicon-carbon composites. This study finds significant limits in current anode technologies and provides feasible techniques for performance improvement.
Wang (2017) underscored the essential requirement for next-generation anode materials that provide enhanced energy density, higher charge-discharge stability, and improved initial coulombic efficiency. Nanostructured materials are emphasized as formidable candidates owing to their capacity to endure repeated cycling and provide efficient ion/electron transport.
In sodium-ion batteries, methods including nanoscale size reduction, surface modification, and composite formulation are acknowledged as effective strategies for performance enhancement. The incorporation of hybrid interfaces and customized material architectures is deemed crucial for the advancement of durable, long-lasting anodes appropriate for extensive energy storage applications.

3.MATERIALS AND METHOD

This research focuses on producing and analyzing nanostructured $NiWO_x$ for its potential as an anode in lithium-ion and sodium-ion batteries. The methodology includes precursor preparation, thermal treatment at 600–700°C, in-depth examination of its structural and surface properties, electrode development, and assessment through electrochemical techniques.

Materials and Reagents

Citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$, analytical grade), Sodium tungstate dihydrate ($Na_2WO_4 \cdot 2H_2O$, ≥99%, Sigma-Aldrich) and Nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$, ≥99%, Merck) were used as precursors without further purification. Deionized (DI) water was employed throughout all experimental procedures.

Synthesis of Nanostructured $NiWO_4$

A sol-gel-aided chemical precipitation technique was used to create nanostructured $NiWO_4$. Under continuous stirring at 60 °C, equimolar (0.1 M) solutions of $Ni(NO_3) \cdot 2H_2O$ and $Na_2WO_4 \cdot 2H_2O$ were combined. To serve as a complexing agent, citric acid was introduced in a 1:1 molar ratio with metal ions. Ammonia solution (NH_4OH) was used to bring the solution's pH to about 7. Dried at 120 °C for 12 h, the resultant gel was then crushed into fine powder. To produce very crystalline $NiWO_4$ nanoparticles, calcination was done in air in a muffle furnace at 700 °C for three hours.

Electrode Fabrication

Mixing 80 weight percent synthetic $NiWO_4$ powder, 10 weight percent polyvinylidene fluoride (PVDF) in N-methyl-2-pyrrolidone (NMP) solvent, and 10 weight percent Super P carbon black to a consistent consistency was the process for creating the working electrode slurry. Following this, the slurry was spread onto aluminium foil for LIBs and copper foil for SIBs. Before hydraulic compression, the electrodes were vacuum-dried for 12 hours at 80°C. The active ingredient reached a final mass loading of about 1.5 mg cm^{-2}.

Electrochemical Measurements

An argon-filled glovebox contained the assembly of CR2032-type coin cells. Made of lithium or sodium metal and a Celgard 2400 separator, the reference and counter electrodes were built. Comprising 5% FEC extra, the sodium-ion battery cell electrolyte was 1 M NaClO in propylene carbonate (PC). Using a 1:1 volume ratio of

dimethyl carbonate (DMC) and ethylene carbonate (EC), a lithium polymer aqueous solution (LPF6) was created by dissolving it in water for the manufacture of lithium-ion batteries (LIBs). During galvanostatic charge-discharge (GCD) cycling within a voltage range of 0.01-3.0 V, several current densities were assessed. A CHI 760E workstation was used to perform electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) in order to assess charge transfer resistance and redox activity.

RESULT AND DISCUSSION

The XRD analysis of $NiWO_4$, as shown in Figure 1, demonstrates that the diffraction patterns shift slightly when the calcination temperature increases from 600°C to 700°C. At 600°C, additional peaks corresponding to WO_3 (marked by asterisks) and NiO (marked by circles) indicate the presence of impurities. However, these impurity peaks disappear at higher calcination temperatures (650°C and above), confirming improved phase purity. Distinct diffraction peaks associated with monoclinic $NiWO_4$ (JCPDS No. 15-0755) are clearly observed across all samples. Refinement of the lattice parameters using the Rietveld method further validates the monoclinic structure, with lattice constants of 4.703 Å, 5.713 Å, and 4.954 Å. The Scherrer formula estimates an average crystallite size of approximately 23 nm, indicating nanoscale crystallites. Grain growth is evident at 700°C, as shown by sharper and more intense peaks, suggesting increased crystallinity and a probable reduction in surface area compared to samples calcined at lower temperatures.

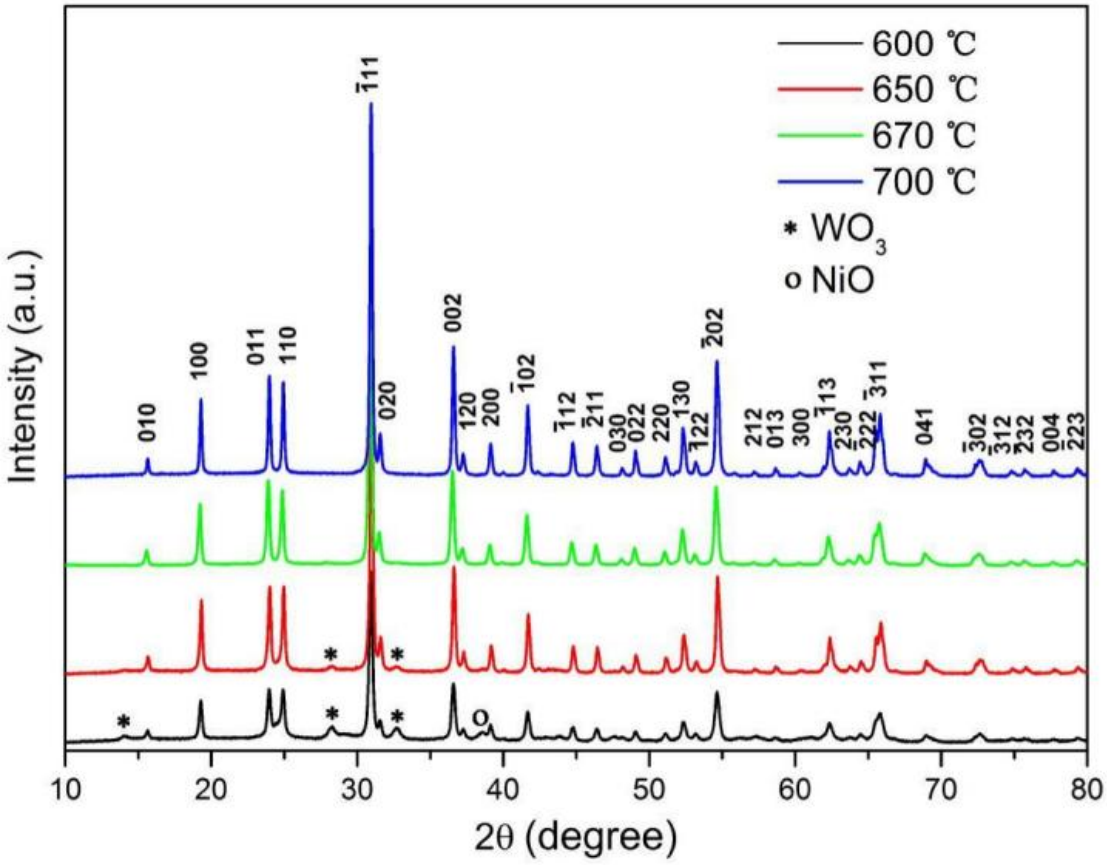

Figure 1. XRD patterns of the NiWO4

Surface Chemistry and Bonding Analysis

The oxidation states of nickel, oxygen and tungsten in the sample were verified by X-ray Photoelectron Spectroscopy (XPS) analysis. The presence of $Ni2^{+}$ ions was evidenced by the spectra, which exhibited distinct peaks with binding energies of 855.8 eV and 873.5 eV, corresponding to the Ni 2p3/2 and Ni 2p1/2 levels, respectively. The presence of tungsten in its $W*^{+}$ oxidation state was evidenced by the pronounced doublets between 35.8 eV and 37.9 eV in the W 4f region.

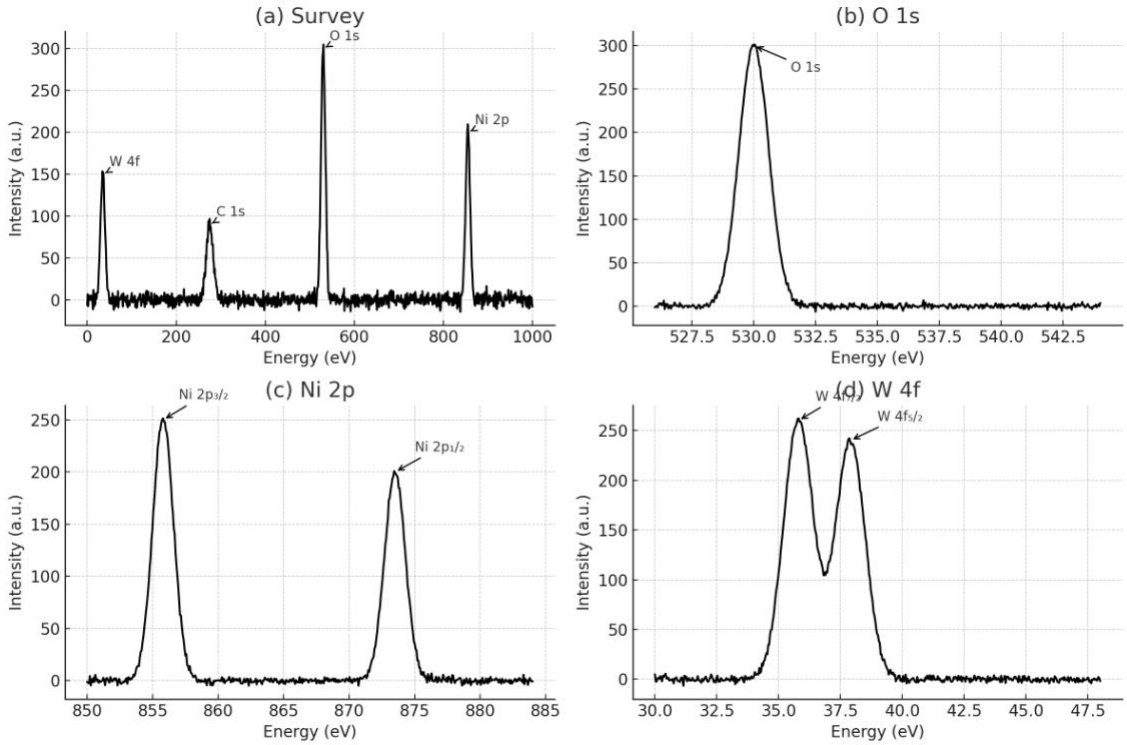

Figure 2. XPS spectra of $NiWO_4$ calcined at 700°C showing (a) survey scan, (b) O 1s, (c) Ni 2p, and (d) W 4f core-level spectra, confirming the presence of Ni^{2+}, W^{6+}, and lattice oxygen

Electrochemical Performance in Lithium-Ion Batteries

Figure 3 illustrates the cyclic voltammetry curves of the $NiWO_4$ electrode within the lithium-ion battery system. In the initial cycle (black curve), the lithiation and delithiation processes of the active material are indicated by pronounced redox peaks at approximately 0.6 V and 1.4 V vs. Li^+/Li, respectively. In the second cycle, seen by the red curve, these peaks are more acute and pronounced. Cycles three and four exhibit more stability (blue and cyan curves, respectively), indicating enhanced electrochemical reversibility and augmented electrode activation. During galvanostatic charge-discharge (GCD) testing at 0.1 A g^{-1}, the conversion-type electrode exhibits the typical irreversibility observed in the initial cycle, with a coulombic efficiency of around 74% and an initial discharge capacity of 832 mAh g^{-1}. A remarkable reversible capacity of 620 mAh g^{-1} persists after 500 cycles under identical conditions, signifying exceptional long-term cycling stability.

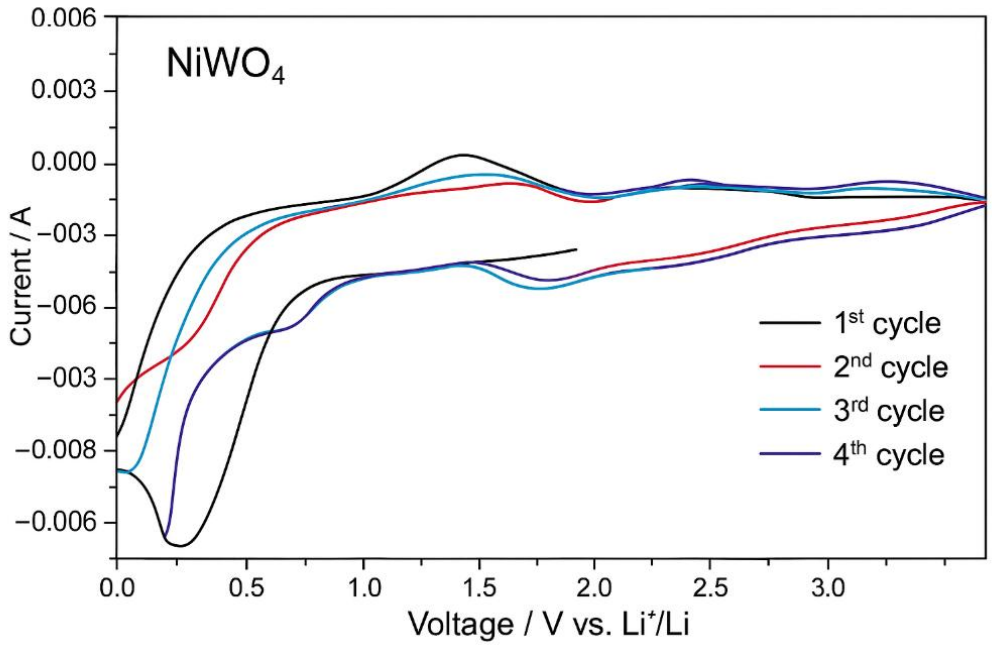

Figure 3. Cyclic voltammetry (CV) curves of $NiWO_4$ electrode in a lithium-ion battery

Electrochemical Performance in Sodium-Ion Batteries

The $NiWO_4$ electrode showed consistent electrochemical performance, with a first-cycle coulombic efficiency higher than 70% and an initial discharge capacity of 521 mAh/g at 0.1 A/g. Capacity retention of 395 mAh/g after 300 cycles demonstrated excellent cycling stability. Despite a drop to 345 mAh/g at 2 A/g, the capacity increased to about 400 mAh/g at 0.1 A/g current density. Galvanostatic intermittent titration method (GITT) and electrochemical impedance spectroscopy (EIS) verified the anticipated transport restrictions, showing a greater internal resistance and reduced Na^+ diffusion coefficients (10^{-12}-10^{-14} cm^2/s) in comparison to Li^+ ions.

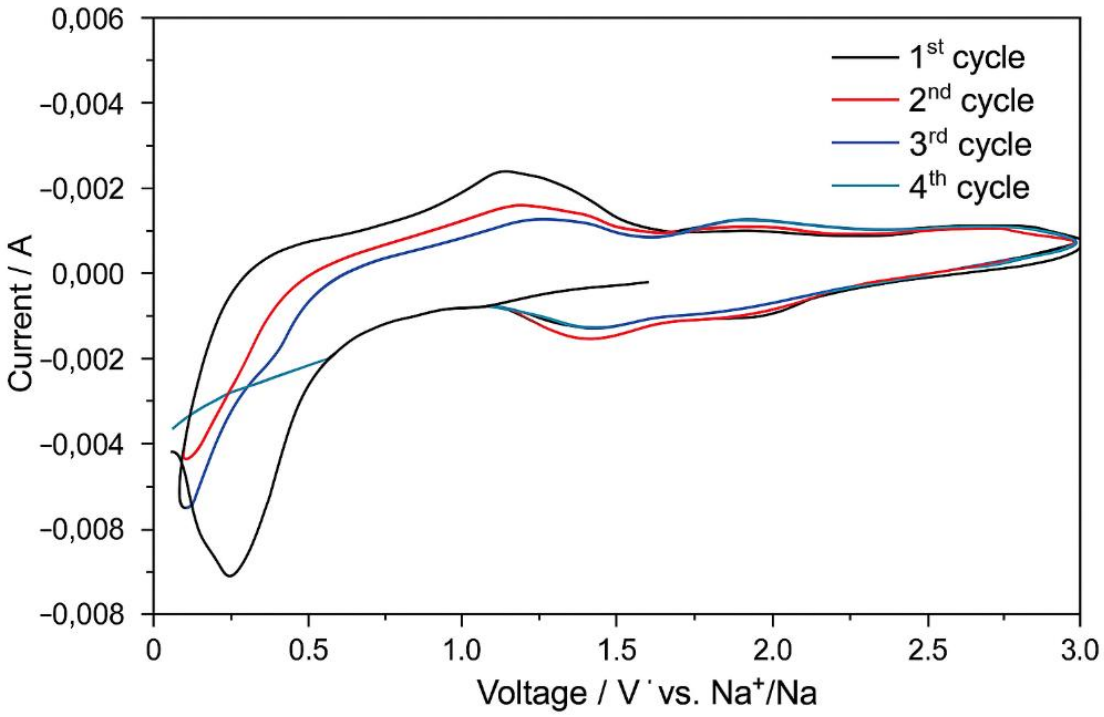

Figure 4. Cyclic voltammetry (CV) curves of $NiWO_4$ electrode in a Sodium-ion battery

Conclusion

This study used a sol-gel assisted chemical precipitation technique to successfully create nanostructured nickel tungstate ($NiWO_4$), which was subsequently heated to 700 °C. It was proven that a highly crystalline monoclinic $NiWO_4$ phase had developed after structural study using XRD and Rietveld refinement. The average size of the crystallites was around 23πm. Surface chemical stability and electrical preparation for redox processes were established through the utilization of XPS measurements, which proved the occurrence of Ni^{3+} and $W*^{+}$ oxidation states. Both sodium-ion and lithium-ion battery topologies demonstrated good performance in the electrochemical experiments conducted with the $NiWO_4$ electrode. The lithium-ion battery's electrode demonstrated outstanding cycling stability by maintaining 620 mAh g^{-1} after 500 cycles and providing a high initial discharge capacity of 832 mAh g^{-1}. The capacity of 395 mAh g^{-1} in sodium-ion systems remained unchanged even after 300 cycles, suggesting outstanding rate performance and capacity recovery. Both complementary gradient-induced transport (GITT) and electrochemical impedance spectroscopy (EIS) verified the low internal resistance and rapid ion movement. Those studies proved that thermally adjusted $NiWO_4$ nanostructures might be used as bifunctional anode materials in the future generation of lithium- and sodium-ion batteries. Scalability and affordability in $NiWO_4$ energy storage devices are made possible by the interplay of thermal treatment, structural stability, and electrochemical performance.

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