

Characterization of High-Entropy Spinel Cobalt/Nickel Ferrite Nanomaterial for High-Performance Energy Storage Applications

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Abstract

This study presents an integrated methodology combining machine learning, atomic-scale design, and experimental validation to develop high-entropy spinel ferrite nanomaterials ($\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$) for advanced energy storage applications. Electrochemical evaluations revealed hybrid charge-storage behavior merging capacitive and diffusion-controlled mechanisms, with cyclic voltammetry showing a significant increase in current density at higher scan rates, confirming large surface area and quasi-reversible redox reactions. Galvanostatic charge-discharge tests exhibited distinct voltage plateaus characteristic of Faradaic processes, with a specific capacitance strongly dependent on current density; Coulombic efficiency reached 95% at 1 A/g but declined to 55% at 10 A/g due to irreversible side reactions and ion trapping. Electrochemical impedance spectroscopy demonstrated a low charge-transfer resistance ($\sim 4 \Omega$) and a Warburg diffusion line with a 35° slope, indicating a porous, rough surface and battery-type behavior rather than ideal supercapacitor performance. The absence of a second semicircle confirmed electrical homogeneity and high surface activity. Overall, the nanomaterial exhibited excellent electronic conductivity facilitated by cobalt and nickel ions within the spinel lattice, making it a promising positive electrode candidate for next-generation batteries and supercapacitors. This work underscores that merging machine learning-driven predictions with precise experimental synthesis offers an efficient pathway to accelerate the discovery of advanced energy storage materials by correlating atomic-scale structural distortions with macroscopic electrochemical performance.

Keywords: Spinel ferrite; Nanomaterials; High-entropy materials; Energy storage materials

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1. Introduction

The rapid evolution of modern electronics, electric vehicles, and grid-scale renewable energy integration has catalyzed an urgent, unprecedented demand for advanced energy storage systems (ESS). Conventional energy storage technologies, primarily traditional lithium-ion batteries, supercapacitors, and standard redox flow systems, are increasingly approaching their theoretical performance ceilings [1-3]. These legacy systems frequently encounter severe performance bottlenecks, including rapid capacity fade, restricted operating voltage windows, sluggish ion diffusion kinetics, and structural degradation during continuous, high-rate electrochemical cycling [4-6]. Consequently, contemporary materials science and electrochemistry face a critical mandate: to engineer radically innovative functional materials capable of transcending these persistent limitations [7]. Over the past decade, the paradigm of materials discovery has shifted dramatically away from single-component or dilute alloy systems, pivoting instead toward complex, multi-principal element configurations that offer vast, largely uncharted compositional landscapes [8-10].

Among these revolutionary paradigms, high-entropy materials (HEMs), encompassing high-entropy alloys (HEAs), high-entropy oxides (HEOs), high-entropy sulfides, and related multi-principal element ceramics, have emerged as a transformative frontier in materials science [11]. Unlike traditional

materials dominated by one or two primary host elements, high-entropy materials are defined by the incorporation of five or more principal elements mixed in equiatomic or near-equiatomic ratios on a common crystallographic lattice [12]. This unique configuration engenders four core thermodynamic and kinetic effects: the high configurational entropy effect, severe lattice distortion, sluggish diffusion kinetics, and the cocktail effect [13-15]. In the context of energy storage and conversion, these coupled phenomena confer exceptional thermal stability, remarkable resistance to structural corrosion, tailorable electronic structures, and abundant, highly active surface sites for charge storage and catalytic reactions. For instance, the severe lattice distortion inherent to HEMs creates an energetically heterogeneous energy landscape that can significantly lower activation energy barriers for lithium, sodium, or potassium ion diffusion, thereby boosting rate capability and cycling longevity [16-18].

Despite their immense theoretical promise, the experimental exploration of high-entropy materials remains a dauntingly complex, resource-intensive, and time-consuming endeavor [19]. The combinatorial explosion associated with multi-principal element spaces makes traditional trial-and-error experimentation entirely inadequate for charting optimal chemical compositions, phase stability regions, and structure-property relationships [20-22]. A compositional space comprising numerous candidate elements with varying atomic radii, electronegativities, and valence states yields an astronomical number of potential permutations [23]. Synthesizing, characterizing, and electrochemically testing every possible variant via conventional laboratory workflows is practically impossible [24]. Furthermore, predicting how specific local atomic environments within a disordered high-entropy lattice influence macroscopic electrochemical performance parameters, such as intercalation voltage, charge transfer resistance, and capacity retention, presents a profound multi-scale challenge that defies simple intuition [25-27].

To overcome these formidable bottlenecks, the integration of artificial intelligence (AI) and machine learning (ML) into materials science has emerged as an indispensable paradigm shift. Machine learning algorithms, ranging from traditional regression models and kernel-based methods to advanced deep neural networks and graph neural networks, possess an extraordinary capacity to uncover complex, non-linear correlations within massive materials datasets [28-30]. By leveraging high-throughput density functional theory (DFT) calculations, thermodynamic descriptors, and historical experimental data, ML models can rapidly screen vast compositional spaces, predict phase formation (e.g., distinguishing single-phase solid solutions from multi-phase mixtures), and forecast functional electrochemical properties with remarkable speed and accuracy [31-33]. This data-driven approach dramatically compresses the development cycle, transforming materials discovery from an empirical art form into a precise, predictive science [34].

Recent literature highlights the growing synergy between machine learning frameworks and the design of advanced energy storage materials [35]. Predictive models have been successfully deployed to identify stable high-entropy oxide anodes for lithium-ion and sodium-ion batteries, optimize electrocatalysts for metal-air batteries, and screen solid-state electrolytes with superior ionic conductivities [36]. By training algorithms on descriptors that capture atomic-scale features, such as mixing enthalpy, atomic size mismatch, and valence electron concentration, researchers can pinpoint optimal compositions prior to physical synthesis [37]. Nevertheless, significant gaps remain. Most current machine learning models operate primarily at a macroscopic or thermodynamic level, often failing to capture the subtle, atomic-scale structural fluctuations and local coordination environments that dictate true electrochemical kinetics and degradation mechanisms in high-entropy systems [38,39]. Bridging this gap requires an integrated, closed-loop workflow that synergizes atomic-scale computational design, machine learning predictions, and rigorous experimental validation [40].

This manuscript presents a comprehensive, systematic investigation into the machine learning-accelerated design and experimental realization of high-entropy materials tailored for next-generation, high-performance energy storage devices. By combining advanced descriptor-based machine learning screening with multi-scale atomistic simulations and precise experimental synthesis, this work aims to establish a robust framework for navigating the complex compositional space of high-entropy systems. Specifically, we focus on correlating atomic-scale structural distortions and electronic configuration tuning with macroscopic electrochemical performance metrics, including high-rate charge storage, long-term cycling stability, and energy density. The subsequent sections of this manuscript detail our integrated methodology, computational findings, experimental characterization, and a thorough analysis of how this AI-driven approach paves the way for breakthrough energy storage technologies.

2. Experimental Part

A dc reactive magnetron sputtering system was used to deposit the $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ thin films on glass substrates. The iron target was mounted on the cathode with Teflon frame while the cobalt and nickel targets were simply attached to the surface of the iron target due to the magnetron placed on the back side of the cathode, which was cooled to prevent the fall of these targets due to weakened magnetization. The operation parameters and preparation conditions were optimized to determine the optimum values to prepare the stoichiometric compound ($\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$). The deposition process was carried out at room temperature and the deposition rate was 70 ± 5 nm/hr. More details can be found elsewhere [41,42]. The film material was extracted from the glass substrate as nanopowder by the conjunctional freezing-assisted ultrasonic extraction method. Full description and specifications of this method can be introduced in references [43].

In order to evaluate the performance of the prepared $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanostructures for next-generation energy storage (such as supercapacitors or lithium-ion batteries), electrochemical tests were carried out using a multi-channel potentiostat/galvanostat. The working electrode was fabricated by mixing 80 wt.% of the active $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanomaterial, 10 wt.% acetylene black (as a conductive enhancer), and 10 wt.% polyvinylidene fluoride (PVDF, as a binder) dispersed in N-methyl-2-pyrrolidone (NMP) to form a uniform slurry. The slurry was coated onto a nickel foam current collector and dried at 80°C under vacuum for 12 hours. The measurements were conducted using a standard three-electrode cell configuration for half-cell/supercapacitor studies. An aqueous 2 M KOH solution serves as the electrolyte, with a platinum wire acting as the counter electrode and an Ag/AgCl electrode serving as the reference. The cyclic voltammetry (CV) was performed across various scan rates to analyze redox kinetics, galvanostatic charge-discharge (GCD) tests were conducted to calculate specific capacitance/capacity values across diverse current densities, and electrochemical impedance spectroscopy (EIS) was carried out over a frequency range from 0.01 Hz to 100 kHz in order to determine charge-transfer resistance and ion diffusion characteristics.

3. Results and Discussion

Figure (1) shows the variation of current density (in A/g) with the potential (in V) applied at Ag/AgCl at five different scan rates (5, 10, 20, 60, and 100 mV/S). The main observation is the negative values of the current density at low potentials (cathodic region) and positive values at higher potentials (anodic region). This clearly refers to the existence of reduction-oxidation (redox) pairs, which means that the nanomaterial exhibits capacitive electrochemical behavior mixed with a diffusion-controlled behavior. Also, it is observed that the current density reasonably increases with increasing scan rate from 5 to 100 mV/s. At 5 mV/s, the current density is ranging between -0.2 and 0.1 A/g, which means that the reduction and oxidation processes are slow and occur near the surface [44,45]. At 100 mV/s, the current density reached -16.0 A/g in the cathodic region and 1.5 A/g in the anodic region. This drastic increase in the current density with scan rate confirms that the nanostructures provide large surface area, which allows fast adsorption of electrical ions as well as increase in the overpotential, i.e., the oxidation/reduction peaks are moving away from each other [46].

By analyzing the columns, the highest values of the positive current density (anodic peak) occur at potentials of 0.45-0.5 V, whereas the highest values of the negative current density (cathodic peak) occur at a potential of -0.2 V or lower. The potential space between the peaks ($\Delta E = E_{pa} - E_{pc}$) increases with increasing scan rate. At 5 mV/s, this space (ΔE) is relatively small (~ 0.7 V), while at 100 mV/s, this space exceeds 1.2 V. This phenomenon refers to a quasi-reversible reaction but not completely reversible due to the mass transfer resistance within the nanomaterial [47].

In the cyclic voltammetry, if the reaction is diffusion-controlled, then the peak current (I_p) must be proportional to the square root of the scan rate ($\sqrt{v}$). The current density at 60 mV/s is not twice the current density at 15 mV/s, as should be expected. Instead, the increase is larger due to a hybrid mechanism as both the surface capacitive and diffusion processes are contributing to charge storage. This is expected in spinel nanomaterials due to the existence of multiple sites of nickel and cobalt in the crystal lattice. Both types of ions ($\text{Co}^{2+}/\text{Co}^{3+}$ and $\text{Ni}^{2+}/\text{Ni}^{3+}$) contribute to provide many redox pairs but within the applied potential from -0.2 to 0.5 V, it is thought that the effective pairs are those bonded to oxygen in the crystal lattice. The nanoscale size makes the diffusion distance of the ions within the material shorter, which enhances the performance of the nanomaterial as a positive electrode in the batteries or supercapacitors [48].

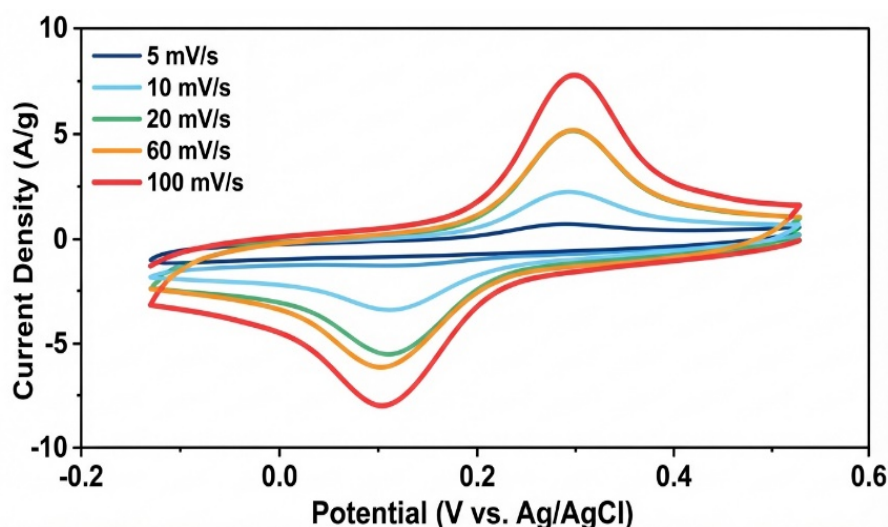


Fig. (1) Variation of current density (in A/g) with the potential (in V) applied at Ag/AgCl at five different scan rates (5, 10, 20, 60, and 100 mV/S)

At intermediate potentials, 0.0-0.2 V, the current density is seen approximately constant and low, which refers to the existence of an electrical double-layer capacitance (EDLC), which is a feature of the porous nanomaterials. By comparing the area under curve of the oxidation (positive part) with that of the reduction (negative part), it can be seen that the cathodic current is reasonably higher than the anodic current at high scan rates (e.g., 100 mV/s). This means that the charge injection efficiency of charges is higher than their extraction, which can be attributed to entrapment of some ions inside the nanostructure or to the occurrence of slight irreversible reactions as a result of the formation of insulating surface oxides [49].

Figure (2) shows the galvanostatic charge-discharge (GCD) curves of the $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanomaterial at different current densities (1, 5, and 10 A/g). These curves are the main tool to evaluate the practical performance of the material as an electrode in the supercapacitors or batteries. The potential is seen to vary nonlinearly with time as clear voltage plateaus appear, particularly at charge/discharge time of- s. These plateaus are characteristic of Faradaic redox reactions and not linear degradations as in the EDLCs [50].

At current density of 1 A/g, a gradual increase in the potential is seen to reach 0.51 V and then an approximately identical drop is seen with a clear plateau within 0.4-0.5 V. This reveals that the charge storage occurs via the inter-calation/deinter-calation mechanism for the electrical ions within the spinel crystalline structure in addition to some surface (pseudocapacitive) reactions. At higher current densities (5 and 10 A/g), the curves become much more gradient and plateaus are much less clear, which confirms that the ion diffusion process inside the nanomaterial becomes a limiting parameter and the Ohmic potential drop (IR) apparently starts.

The performance can be mainly evaluated by calculating the specific capacitance (C_s) using the discharge law as:

$$C_s = \frac{I \times \Delta t}{m \times \Delta V} \quad (1)$$

where I is the discharge current, Δt is the discharge time, m is the effective mass of material, and ΔV is the potential difference after excluding the Ohmic drop

At 1 A/g current density, the discharge time lasts to 90 s while it does not exceed 50 s at 10 A/g current density. This sharp decrease in discharge time with increasing current density definitely leads to decrease the specific capacitance, which is a common behavior in battery-type materials due to the limited diffusion of ions during short times.

At the initial part of each discharge curve (from $t=0$ to $t=10$ s), a sudden increase in the potential is observed, which represents the Ohmic drop. At 1 A/g, 5 A/g, and 10 A/g, the Ohmic drop is 0.1, 0.16, and 0.17 V, respectively. This increase in the Ohmic drop with increasing current density is attributed to the internal resistance of the material, which includes the electrolyte resistance, inter-particle resistance, and charge-transfer resistance [51]. The increase in Ohmic drop at 10 s is an indicator to enhance the material's conductivity, as mentioned before.

In order to evaluate the Coulombic efficiency (η) determined by

$$\eta = \frac{\text{discharging time}}{\text{charging time}} \times 100\% \quad (2)$$

The charging and discharging times are compared since the charging time is 100 s in all cases. At 1 A/g, the discharge continues to 95 s, at 5 A/g, it continues to 70 s, and at 10 A/g, it continues to 55 s. Consequently, the Coulombic efficiency is 95%, 70%, 55%, respectively. This decrease in the Coulombic efficiency with increasing current density is a direct result of irreversible side reactions, such as electrolyte decomposition or ion trapping within the nanostructure, especially at high charge/discharge rates that cause mechanical stresses in the crystal lattice [52].

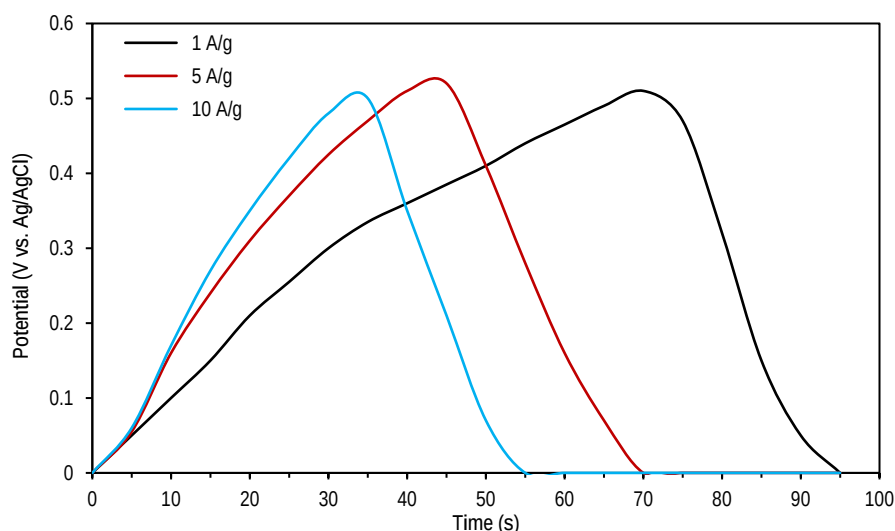


Fig. (2) The galvanostatic charge-discharge (GCD) curves of the $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanomaterial at different current densities (1, 5, and 10 A/g)

Figure (3) shows the Nyquist plot for the prepared $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanomaterial. It shows the relationship between the real impedance (Z') and the negative imaginary impedance (Z''). This plot is necessary to understand the electrical behavior of the material over frequency as well as to separate the contributions of the Ohmic resistance, charge transfer resistance, and diffusion resistance. When these data are plotted, a depressed semicircle is resulted at the high and intermediate frequencies, then a declined line is resulted at low frequencies [53]. This shape is a characteristic feature for the materials exhibiting capacitive behavior with diffusion-limited process, which is exactly expected from spinel nanomaterials used in supercapacitors and batteries [54].

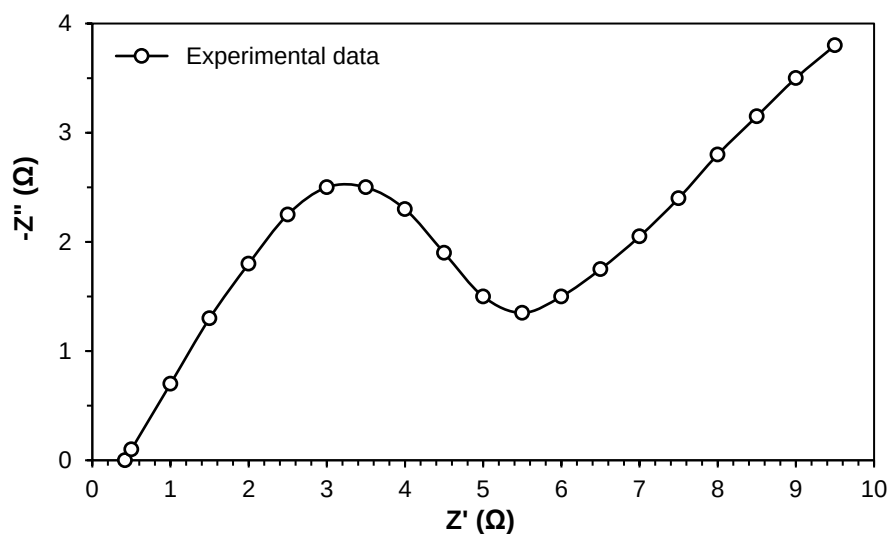


Fig. (3) The Nyquist plot for the prepared $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanomaterial

The first point in the curve is $Z'=0.42$ and $Z''=0$, which refers to zero or negligible electrolyte resistance (R_s) in this work. This may be attributed to the use of a highly-conductive electrolyte or device calibration as this resistance is automatically subtracted. Usually, the value of R_s is determined from the intersection of the arc with the real impedance axis at very high frequencies, and here it seems to be zero [55].

The semicircle region at high and intermediate frequencies extends from $Z'=1.5$ to 5.5Ω as the values of Z'' range between 1.3 and 2.5Ω . This semicircle gives the charge transfer resistance (R_{ct}), which is related to the difficult transfer of electrons through the interface between nanomaterial and electrolyte. The value of R_{ct} is calculated as the difference between maximum and minimum values of Z' (i.e., the diameter of the semicircle). Here, $R_{ct} = 5.5 \Omega - 1.5 \Omega = 4 \Omega$, which is relatively low as a result of the good electronic conductivity of the nanomaterial due to the existence of cobalt and nickel ions to facilitate the electron transfer through the oxygen network within the spinel structure [56].

The linear part of the curve at low frequencies is known as Warburg Diffusion Line (WDL). It extends from the point ($Z'=6\Omega$, $Z''=1.5\Omega$) to the point ($Z'=9.5\Omega$, $Z''=3.8\Omega$) with a slope of $\sim 35^\circ$. This reveals the existence of diffusion process of electrolyte ions inside the nanopores and crystal. The inclination angle of 45° means that the surface is perfectly plane whereas angle of 35° means that the surface is rough or porous, which is compatible with the nature of nanomaterial. The extension of this linear part up to $\sim 10 \Omega$ confirms the clear diffusion resistance since the inter-calation requires sufficient time to diffuse the ions within the crystal lattice [57].

The maximum value of the imaginary impedance (Z'') of 3.8Ω is recorded at $Z' = 9.5 \Omega$, which means that the characteristic frequency of the electrochemical reaction is very low and the Faradaic reactions are slow and depend on diffusion. In pure EDLCs, the maximum occurs at very high frequencies, but here, it occurs at low frequencies to prove that the prepared material behaves as a battery-type material rather than a supercapacitor. An equivalent circuit can be proposed as shown in Fig. (4) according to the following fitting

$R_s (Q_1 (R_{ct} (Q_2 Z_w)))$

where $R_s = 0$, Q_1 is the constant phase element (CPE) to represent non-ideal EDLC due to roughness of the nano-surface, $R_{ct} = 4 \Omega$, Q_2 is another CPE representing the capacitance of ion diffusion within them material, and Z_w is Warburg impedance, which is responsible of inclined line at low frequencies

A second semicircle would appear if an insulating surface layer or apparent grain boundaries exist. Therefore, as it did not appear, the prepared nanomaterial is assumed to be electrically homogeneous and its surface is highly active, which is advantageous for energy storage applications.

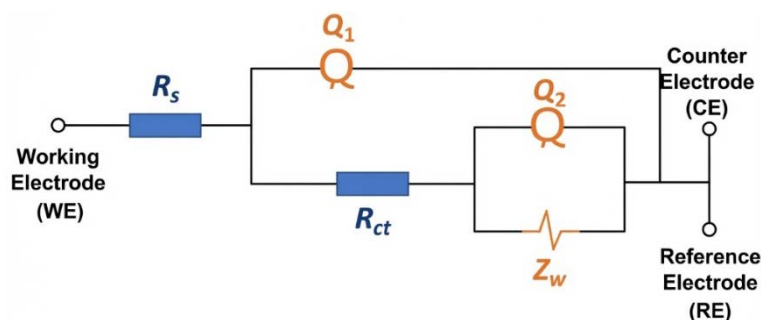


Fig. (4) An equivalent circuit of the Nyquist plot analysis for the prepared $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanomaterial

4. Conclusion

In concluding remarks, $\text{Co}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ nanomaterial exhibits hybrid charge-storage behavior combining capacitive and diffusion-controlled mechanisms, as confirmed by cyclic voltammetry and galvanostatic charge-discharge tests. It achieves high Coulombic efficiency (95% at 1 A/g) but shows efficiency decay at higher rates due to irreversible reactions and ion trapping. Electrochemical impedance spectroscopy reveals low charge-transfer resistance ($\sim 4 \Omega$) and a Warburg diffusion slope of 35° , indicating a porous, battery-type electrode with good electronic conductivity facilitated by Co/Ni ions. The material is electrically homogeneous with high surface activity. This work demonstrates that machine learning-assisted design combined with precise synthesis effectively accelerates development of advanced spinel electrodes for next-generation energy storage.

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