Preparation and Characterization of a Ni-Co-Ce Coordination Framework Coated Nickel Foam Electrode for Supercapacitor Applications

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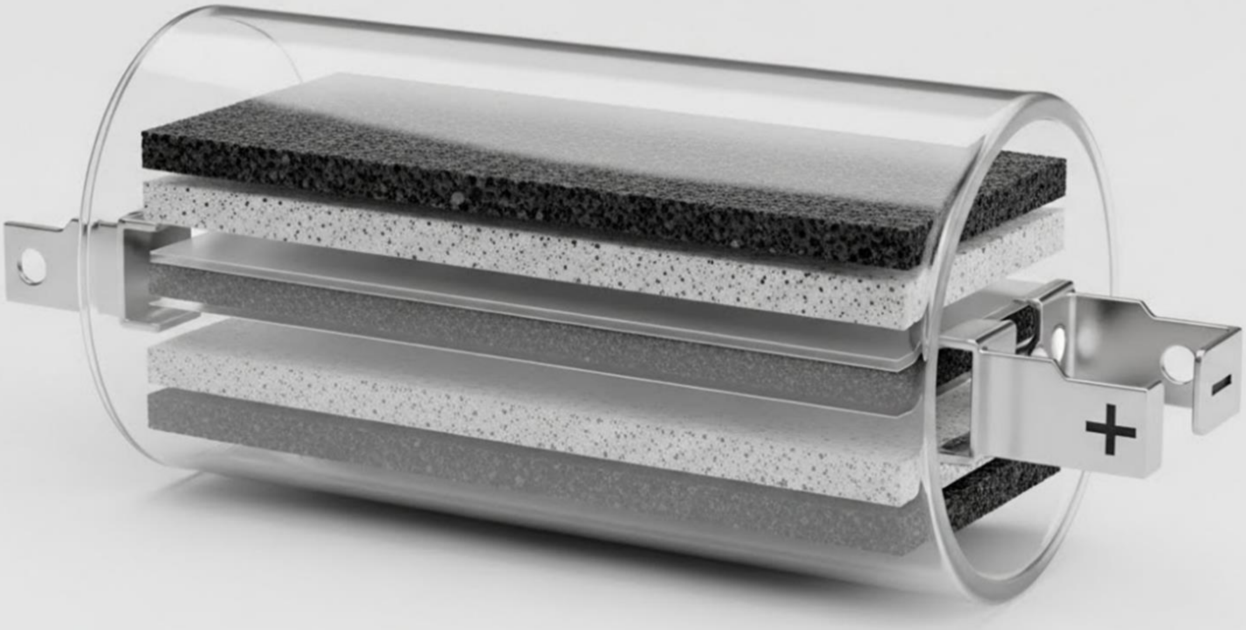
Editor's note: The performance of supercapacitors is heavily influenced by the electrode materials used. These materials should exhibit excellent electrical conductivity, a large surface area, and a high number of electroactive sites. Borhani et al. reported the synthesis of a trimetallic Ni-Co-Ce coordination compound using a hydrothermal synthesis method, which serves as a coating for nickel foam-based electrodes in supercapacitors. This new coating demonstrated stable electrochemical performance, achieving an increase in specific capacitance from 0.74 F/g to 2.6 F/g over multiple cycles.

doi: 10.22034/jams.2025.210154

How to cite: Y. Borhani et al. *Journal of Applied Material Science*, **2025**, *1*, 210154.



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Journal of Applied Material Science **2025**, *1*, 210154
How to cite: Y. Borhani et al., *Journal of Applied Material Science*, **2025**, *1*, 210154.
Journal homepage: **jams.hsu.ac.ir**

Original Research

Preparation and Characterization of a Ni-Co-Ce Coordination Framework Coated Nickel Foam Electrode for Supercapacitor Applications

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Abstract

The rapid development of portable electronics, electric vehicles, and renewable energy systems has heightened the demand for high-performance energy storage devices. In this study, a trimetallic Ni–Co–Ce coordination framework (CF) was successfully synthesized via a simple hydrothermal method and directly deposited onto nickel foam as an electrode for supercapacitor applications. Structural and morphological characterization revealed a hierarchical architecture of oval-shaped microcrystals decorated with chestnut-like protrusions, providing abundant electroactive sites and facilitating efficient ion transport. Electrochemical measurements, including cyclic voltammetry and galvanostatic charge-discharge tests, demonstrated moderate capacitive behavior with specific capacitances increasing from 0.74 F/g to 2.6 F/g over successive cycles. Although Coulombic efficiency decreased from 23.5% to 11.6%, the incorporation of Ce enhanced structural stability and improved electron transport within the framework. These results suggest that Ni-Co-Ce coordination frameworks are promising candidates for supercapacitor applications, and further optimization of material composition and cycling conditions may lead to improved energy storage performance.

Keywords: Coordination Frameworks; Coordination Polymers; Nickel Foam Electrodes; Supercapacitors; Hydrothermal Synthesis.

1. Introduction

The rapid growth of portable electronics, electric vehicles, and renewable energy technologies has created an increasing demand for high-performance energy storage devices [1]. Supercapacitors, also known as electrochemical capacitors, have attracted significant attention due to their high-power density, long cycle life, and fast charge–discharge capability [2]. Unlike batteries, which store energy through slow faradaic reactions, supercapacitors combine electrostatic and

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Received 24 October 2025
Revised 12 December 2025
Accepted 14 December 2025
Available online 18 December 2025

https://doi.org/10.22034/jams.2025.210154

faradaic charge storage mechanisms, offering rapid energy delivery and excellent cycling stability [3]. The performance of supercapacitors is highly dependent on the electrode materials, which must exhibit high electrical conductivity, large surface area, and abundant electroactive sites [4].

Coordination frameworks (CFs), a broader class that includes coordination frameworks (CFs), have emerged as promising materials for energy storage applications due to their tunable porosity, structural diversity, and the possibility to incorporate multiple metal centers [5]. Among transition-metal-based systems, Ni-Co coordination frameworks have demonstrated outstanding electrochemical properties owing to the synergistic redox activity of nickel and cobalt ions [6]. The introduction of a third metal, such as cerium (Ce), into the Ni-Co coordination network can further enhance structural stability, facilitate electron transfer, and improve specific capacitance.

Recent studies have shown that Ni-Co-based coordination frameworks (CFs) exhibit high capacitance, good rate capability, and long-term cycling stability [7]. However, despite their promising electrochemical properties, these materials often suffer from low intrinsic electrical conductivity and limited structural stability during repeated charge-discharge cycling, which can restrict their practical applicability [8]. Incorporating Ce into the Ni-Co coordination network can alleviate these limitations by enhancing electron transport pathways and reinforcing the structural integrity of the framework through continuous and repeated cycling [9].

In this study, a trimetallic Ni-Co-Ce CF was synthesized via a simple hydrothermal method and deposited onto nickel foam as an electrode for supercapacitor applications. The structural, morphological, and electrochemical properties of the material were systematically investigated using FTIR, Raman, TEM, EDX, and electrochemical measurements, including charge-discharge cycling and specific capacitance tests.

2. Experimental

2.1. Materials

Nickel nitrate ($Ni(NO_3)_2 \cdot 6H_2O$), cobalt nitrate ($Co(NO_3)_2 \cdot 6H_2O$), cerium nitrate ($Ce(NO_3)_3 \cdot 6H_2O$), 1,3,5-benzenetricarboxylic acid (PTA, trimesic acid) as the organic linker, dimethylformamide (DMF), and sodium hydroxide (NaOH, 0.4 M) were purchased from commercial suppliers and used without further purification. Nickel foam (1 × 3 cm pieces) used as the substrate for electrode preparation was obtained from Redox Company. Polyvinylidene fluoride (PVDF) and N-methyl-2-pyrrolidone (NMP) were used as binder and solvent, respectively. Ethanol and deionized water were used for washing and cleaning purposes.

2.2. Hydrothermal Synthesis of Ni-Co-Ce Coordination Framework

The trimetallic Ni-Co-Ce CF was synthesized using a hydrothermal route. In a typical procedure, 0.083 g of PTA (1,3,5-benzenetricarboxylic acid, trimesic acid) as the organic linker, 0.0170 g of $Ni(NO_3)_2 \cdot 6H_2O$, 0.0210 g of $Co(NO_3)_2 \cdot 6H_2O$, and 0.0094 g of $Ce(NO_3)_3 \cdot 6H_2O$ were added to 10 mL of DMF under magnetic stirring for 30 minutes. During stirring, 1 mL of 0.4 M NaOH solution was added dropwise to the mixture. After complete mixing, the resulting solution was transferred into a Teflon-lined stainless-steel autoclave and maintained at 100°C for 12 hours. The product was then divided into three Falcon tubes and washed four times with a water–ethanol mixture, followed by three washes with deionized water under ultrasonication. Finally, the collected precipitate was dried in a watch glass at 50°C for 6 hours in an oven before further use.

2.3. Preparation of Ni-Co-Ce CF-Based Electrode

Nickel foam was used as a conductive substrate for the deposition of the synthesized Ni–Co–Ce CF. Prior to coating, the nickel foam pieces (1 × 3 cm) were thoroughly cleaned to remove surface impurities and enhance adhesion. The cleaning procedure involved ultrasonic washing in acetone for 15 minutes, immersion in a solution of 10 mL distilled water and 1 mL HCl for 15 minutes, followed by ultrasonic cleaning in an ethanol–water mixture for another 15 minutes. Finally, the foams were dried in an oven at 50–60°C for 30 minutes.

The electrode paste was prepared by mixing 80 wt% of the Ni-Co-Ce CF as the active material, 10 wt% polyvinylidene fluoride (PVDF) as the binder, and 10 wt% conductive carbon black. The binder solution was obtained by dissolving PVDF in N-methyl-2-pyrrolidone (NMP) with a weight ratio of 1.25% PVDF to 98.75% NMP. The components were thoroughly mixed to form a uniform and viscous paste suitable for coating.

The prepared paste was uniformly applied onto both sides of the cleaned nickel foam using tweezers to ensure good coverage and electrical contact. The coated samples were then dried in an oven at 50°C for 3 hours to remove residual solvent and achieve firm adhesion. The resulting Ni-Co-Ce CF-based nickel foam electrodes were directly employed for structural and electrochemical characterizations.

2.4. Measurements

The morphological and compositional properties of the synthesized Ni-Co-Ce CF were systematically investigated. Scanning electron microscopy (SEM, MIRA3-TESCAN) was employed to observe the surface morphology, particle size, and uniformity of the CF coating on the nickel foam substrate. Energy-dispersive X-ray spectroscopy (EDX, TESCAN MIRA 3 LMU) was used to determine the elemental composition and to confirm the successful incorporation and homogeneous distribution of Ni, Co, and Ce elements within the framework. Furthermore, the electrochemical performance of the prepared electrodes was evaluated through galvanostatic charge-discharge (GCD) tests and specific capacitance measurements, using a RADSTAT 30, which provided insight into the charge storage capability, reversibility, and stability of the Ni–Co–Ce CF electrodes for potential applications in supercapacitors. Electrochemical measurements were carried out in a three-electrode system using 1 M KOH as the electrolyte. A Ni-Co-Ce CF-coated nickel foam served as the working electrode, with graphite as the reference electrode and platinum as the counter electrode. Cyclic voltammetry (CV) was performed within a potential window of 0-0.5 V at scan rates ranging from 5 to 50 mV s^{-1}, while galvanostatic charge-discharge (GCD) tests were conducted at current densities of 0.05-2 mA g^{-1}. These conditions ensure reproducibility and consistent evaluation of the electrode's electrochemical performance.

3. Results and discussion

3.1. Structural Characterization via X-ray Diffraction (XRD)

The crystalline structure of the synthesized Ni-Co-Ce CF was analyzed using XRD, and the diffraction pattern is presented in Figure 1. The main characteristic peaks are observed at 2θ values of 22.3°, 28.7°, 33.2°, 47.9°, 53.3°, and 56.5°. These sharp and well-defined peaks confirm the successful formation of a highly crystalline trimetallic Ni-Co-Ce CF framework. The presence of six main reflections indicates a well-ordered crystalline structure, reflecting the uniform incorporation of Ni, Co,

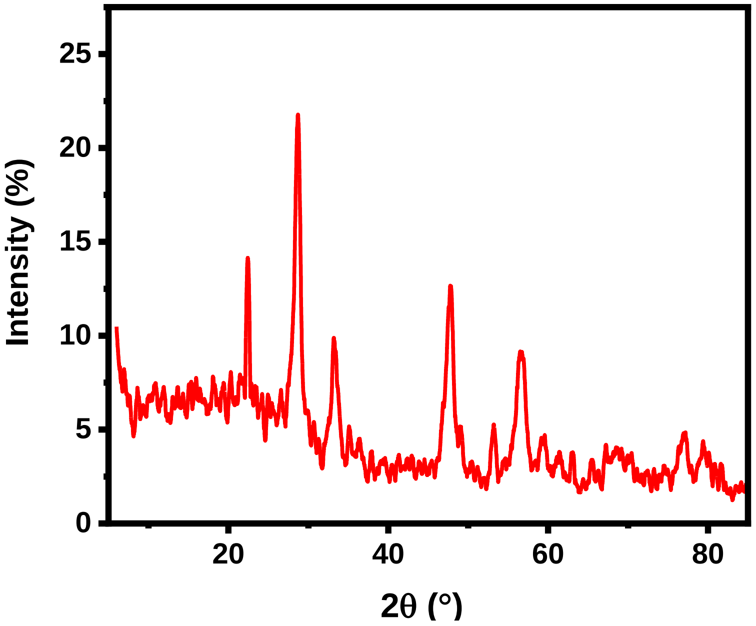

Figure 1. XRD pattern of the as-synthesized Ni-Co-Ce CF, confirming its crystalline structure.

and Ce within the CF. It is noteworthy that the formation of CF alters the local coordination environment of the metal ions compared to their pure metal or oxide forms. Coordination with organic linkers results in the observed diffraction pattern, which is consistent with reported Ni-Co-based CFs and the expected contribution of Ce. The absence of peak broadening suggests a high degree of crystallinity, which supports the structural stability of the synthesized CF. Overall, the XRD analysis confirms that the synthesized Ni-Co-Ce CF possesses a stable and highly crystalline framework, suitable for application as an electrode material in supercapacitors.

3.2. Morphological and Elemental Analysis

The surface morphology of the synthesized Ni-Co-Ce CF was examined using SEM to gain insight into its structural characteristics relevant to electrochemical performance. As shown in Figure 2, the material exhibits a dual hierarchical morphology. The primary structure consists of relatively large, well-defined oval-shaped crystals with smooth and dense surfaces, typically in the range of 1-3 μm. This morphology reflects controlled crystal growth and strong coordination interactions between the metal centers and the organic linker during hydrothermal synthesis. In addition, the surfaces of these oval crystals are partially decorated with secondary chestnut-like protrusions, which are significantly smaller, typically 200-800 nm in size. These spiky nano-features increase the overall surface roughness and create a more open and interconnected architecture, which is beneficial for electrolyte penetration and ion transport. The presence of this hierarchical arrangement enables a greater number of electrochemically active sites while facilitating rapid

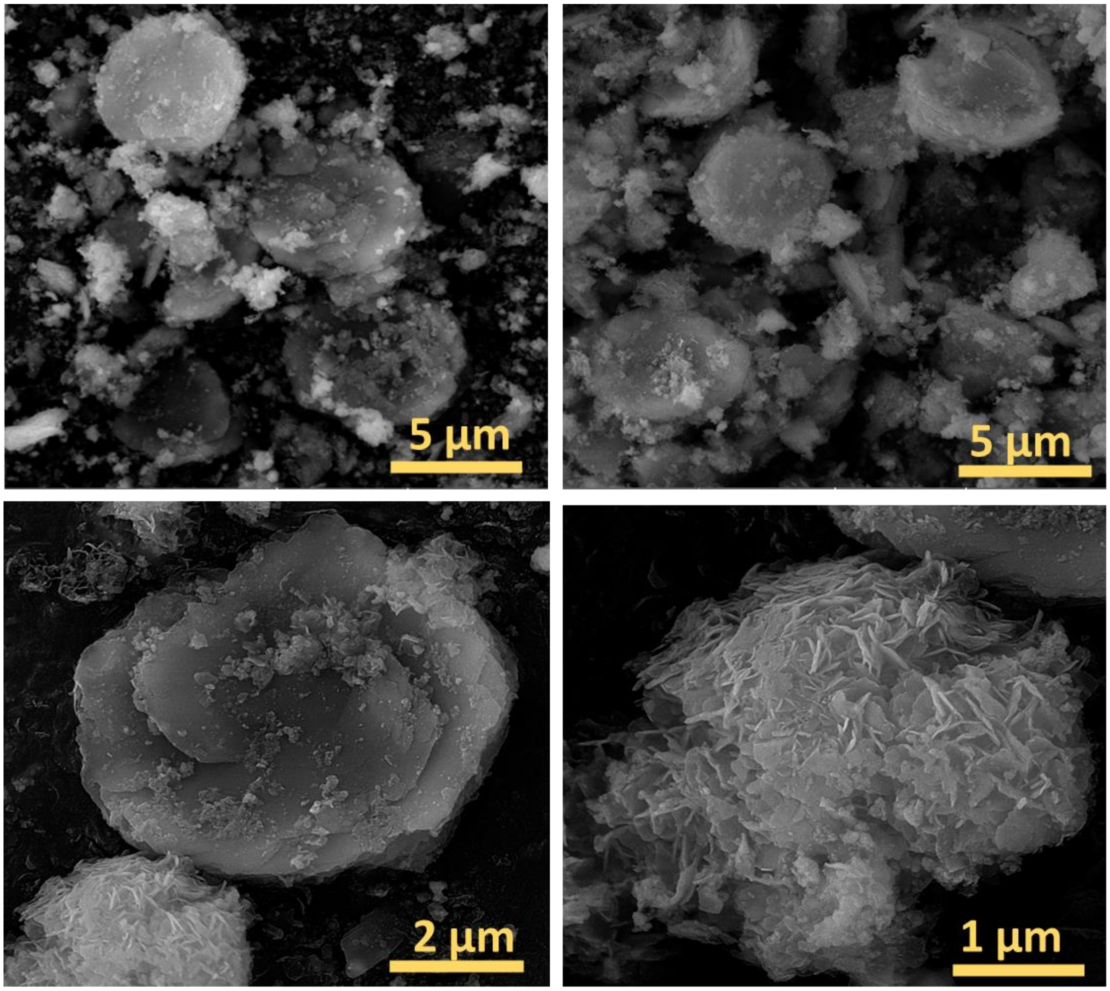

Figure 2. SEM images of the synthesized Ni-Co-Ce CF at different magnifications.

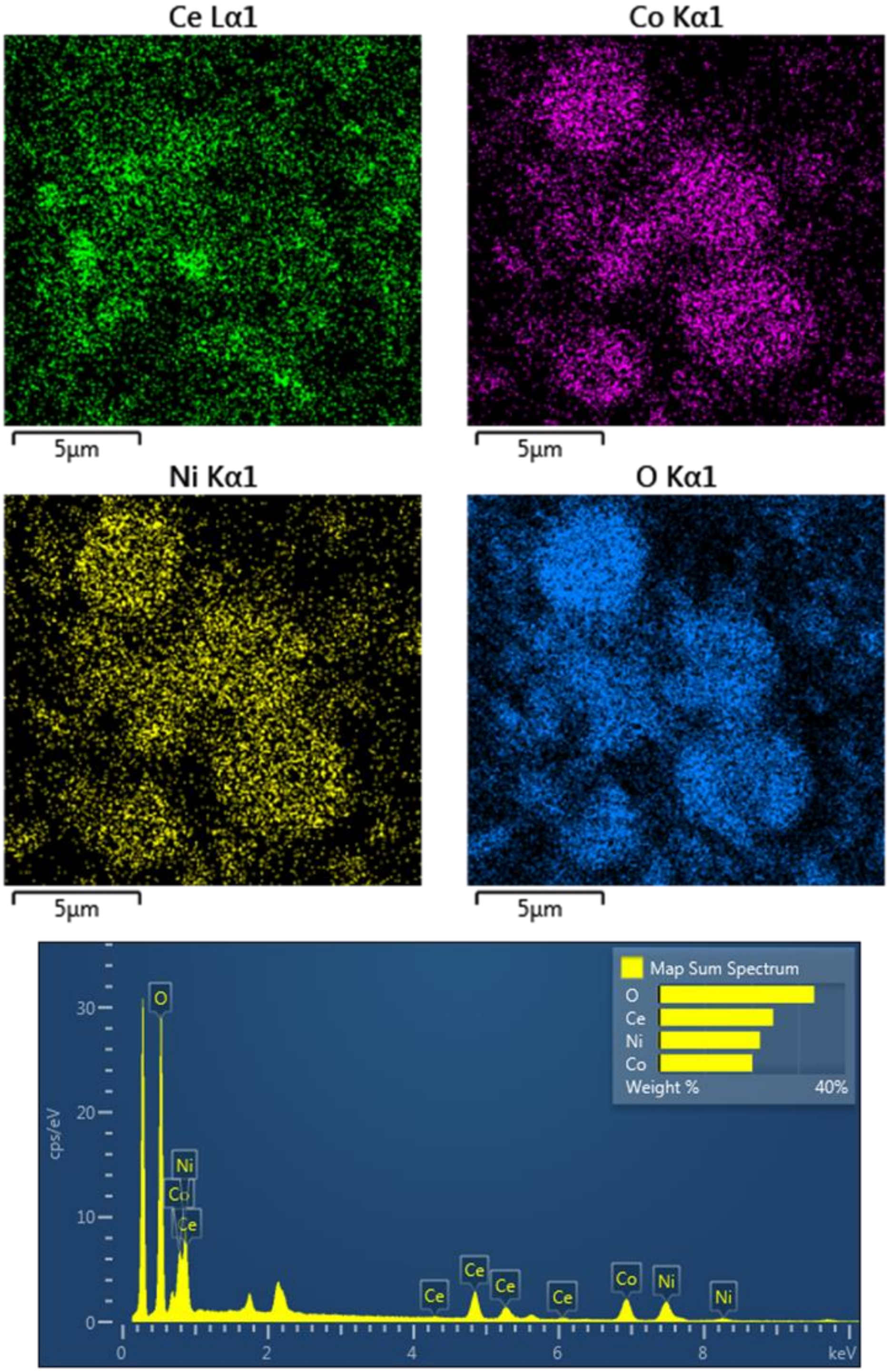

Figure 3. EDX spectrum and elemental mapping of Ni-Co-Ce CF, showing uniform distribution of Ni, Co, Ce, and O, confirming successful Ce incorporation and a hierarchical structure with abundant electroactive.

electrolyte ion diffusion and efficient charge transfer pathways throughout the structure. This morphology was previously reported for bimetallic Ni-Co metal organic frameworks by Jihwa Hong et al [10]. However, the introduction of Ce into the Ni-Co metal node configuration results in noticeable modifications to crystal evolution. The Ce^{3+}/Ce^{4+} redox couple promotes defect formation and heterogeneous nucleation, which enhances the density and distribution of the spiky surface features. This contributes to a larger exposed electroactive area and improves charge transport pathways within the electrode.

The elemental composition of the synthesized Ni-Co-Ce CF was analyzed using EDX, and the results are presented in Figure 3. The spectrum confirms the presence of Ni, Co, Ce, and O, in addition to C from the organic linker, verifying the successful incorporation of Ce into the Ni-Co CF framework. Quantitatively, the weight percentages are 21.82% Ni, 20.15% Co, 24.62% Ce, and 33.40% O. The high atomic fraction of oxygen is consistent with the presence of the organic ligands. Elemental mapping further reveals a uniform distribution of Ni, Co, and Ce throughout the CF structure, consistent with SEM observations of homogeneous oval-shaped microcrystals decorated with chestnut-like protrusions. The even dispersion of these metal components, combined with the hierarchical morphology, ensures a high density of electrochemically active sites. The incorporation of Ce not only contributes additional redox-active centers but also appears to stabilize the CF framework, supporting structural integrity and providing an open architecture favorable for supercapacitor applications.

3.3. *Electrochemical Analysis of the Ni-Co-Ce CF Electrode*

The electrochemical performance of the synthesized Ni-Co-Ce CF electrodes was systematically evaluated using cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) measurements. The CV curves (Figure 4) exhibit distinct redox peaks, indicating reversible faradaic reactions associated with the Ni and Co species in the electrode. The CV curves of the Ni-Co-Ce CF electrode exhibit quasi-rectangular shapes with broad redox peaks, indicating a combination of electric double-layer capacitance and pseudocapacitive behavior. Scans 1 and 2 show relatively lower peak currents, suggesting that the electrode requires additional cycles to reach its maximum electrochemical activity. In contrast, Scans 3 and 4 display higher current densities and more pronounced redox peaks, reflecting enhanced charge storage resulting from improved ion accessibility and an optimized electrode structure. Among the four scans, Scan 4 demonstrates the highest current response, reaching peak currents of approximately 1.64 mA, significantly higher than those observed in Scans 1–3. This enhancement indicates more efficient charge storage capability and improved electrochemical activity for this particular sample. The gradual increase in peak current with successive scans suggests favorable kinetics and enhanced ion transport within the CF structure.

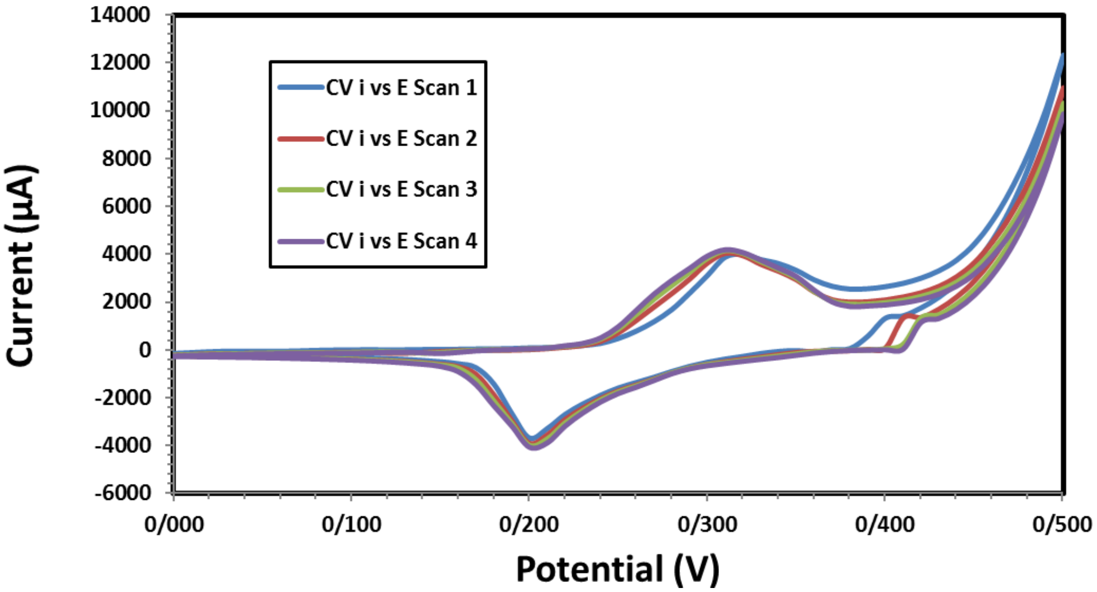

Figure 4. Cyclic voltammetry (CV) curves of the Ni-Co-Ce CF electrode at four successive scans.

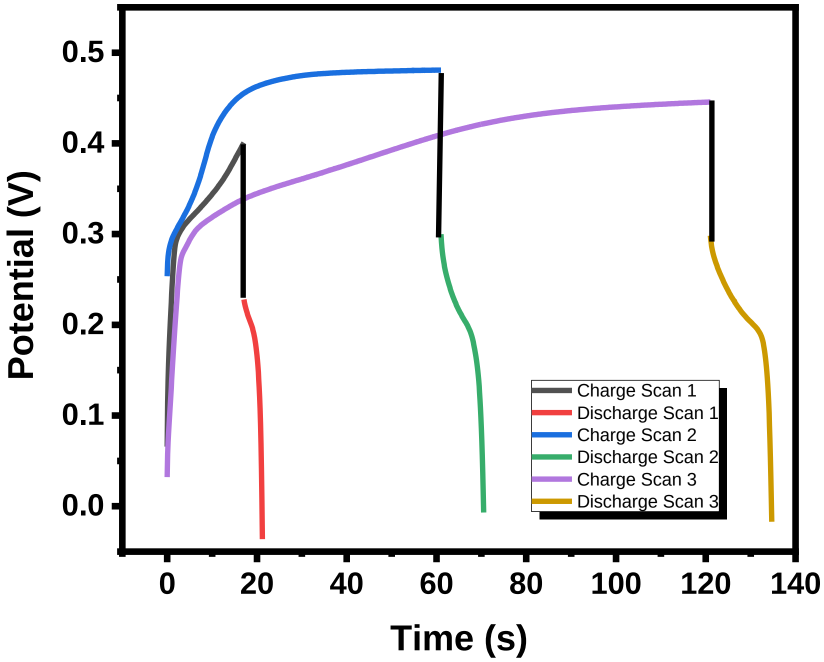

Figure 5. Galvanostatic charge-discharge (GCD) curves of the synthesized Ni-Co-Ce CF-based supercapacitor at three different scan rates.

The galvanostatic charge-discharge (GCD) results of the Ni-Co-Ce CF electrode are presented in Figure 5, with the corresponding specific capacitance and Coulombic efficiency summarized in Table 1. The charge-discharge curves were used to evaluate the electrode's specific capacitance and energy storage efficiency, considering an active material mass of 0.15 g.

The specific capacitance (C_S) was calculated using the equation:

$$C_S = \frac{I.\Delta t}{m.\Delta V} \quad (1)$$

where I is the discharge current (A), Δt is the discharge time (s), m = 0.015 g is the mass of the active material, and $\Delta V \approx$ 0.5 V is the voltage window.

The charge-discharge analysis of the electrode demonstrates a progressive increase in both charge and discharge times across the three scans. Specifically, the discharge time increases from 4 s in Scan 1 to 14 s in Scan 3, indicating a gradual enhancement in the electrode's ability to store charge. Correspondingly, the specific capacitance (Cs) rises from 0.74 F/g to 2.6 F/g, reflecting improved charge storage with longer cycling.

However, the Coulombic efficiency shows a decreasing trend, dropping from 23.5% in Scan 1 to 11.6% in Scan 3. This decline suggests that although the electrode can store more charge at longer charge-discharge durations, energy losses during cycling increase, possibly due to resistive losses or side reactions. Overall, these results indicate that the electrode exhibits moderate capacitive behavior with relatively low efficiency, and optimization of the electrode material or electrolyte may be necessary to enhance reversibility and energy retention.

Table 1. Charge-discharge data, specific capacitance, and Coulombic efficiency of the Ni-Co-Ce CF electrode over three successive scans

Scan	Charge Time (s)	Discharge Time (s)	Specific Capacitance Cs (F/g)	Coulombic Efficiency (%)
1	17	4	0.74	23.5
2	61	9.5	1.76	15.6
3	121	14	2.6	11.6

4. Conclusions

In summary, a trimetallic Ni-Co-Ce CF was successfully synthesized and employed as an electrode for supercapacitor applications. SEM and EDX analyses confirmed a hierarchical micro/nano-structured morphology with uniform distribution of Ni, Co, and Ce, providing numerous electroactive sites and favorable pathways for ion diffusion. Electrochemical studies revealed that the electrode exhibits increasing specific capacitance with prolonged cycling, reaching 0.28 F/g, though Coulombic efficiency decreased, indicating energy losses during charge–discharge cycles. The introduction of Ce contributed to enhanced structural stability and electron transport, mitigating some limitations of conventional Ni-Co CFs. Overall, the study demonstrates the potential of trimetallic CF electrodes for high-performance supercapacitors and provides a foundation for further optimization to achieve higher efficiency and long-term stability.

Conflict of Interest

The authors declare no conflict of interest.

References

1. A. Aghmadi and O.A. Mohammed. Energy storage systems: technologies and high-power applications. *Batteries*, **2024**, *10*, 141.
2. Y.M. Volfkovich. Electrochemical supercapacitors (a review). *Russian Journal of Electrochemistry*, **2021**, *57*, 311.
3. S.Y. Attia, et al. Supercapacitor electrode materials: addressing challenges in mechanism and charge storage. *Reviews in Inorganic Chemistry*, **2022**, *42*, 53.
4. P. Forouzandeh, V. Kumaravel, and S.C. Pillai. Electrode materials for supercapacitors: a review of recent advances. *Catalysts*, **2020**, *10*, 969.
5. H. Laeim, et al. Porosity tunable metal-organic framework (MOF)-based composites for energy storage applications: recent progress. *Polymers*, **2025**, *17*, 130.
6. S. Li, et al. Transition metal-based bimetallic MOFs and MOF-derived catalysts for electrochemical oxygen evolution reaction. *Energy & Environmental Science*, **2021**, *14*, 1897.
7. X. Hang, et al. From Co-MOF to CoNi-MOF to Ni-MOF: a facile synthesis of 1D micro-/nanomaterials. *Inorganic chemistry*, **2021**, *60*, 13168.
8. G. Xu, C. Zhu, and G. Gao. Recent progress of advanced conductive metal–organic frameworks: precise synthesis, electrochemical energy storage applications, and future challenges. *Small*, **2022**, *18*, 2203140.
9. Z. Hu, Y. Wang, and D. Zhao. The chemistry and applications of hafnium and cerium (IV) metal–organic frameworks. *Chemical Society Reviews*, **2021**, *50*, 4629.
10. J. Hong, S.-J. Park, and S. Kim. Synthesis and electrochemical characterization of nanostructured Ni-Co-MOF/graphene oxide composites as capacitor electrodes. *Electrochimica Acta*, **2019**, *311*, 62.
