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ADVANCED ELECTROCHEMICAL CHARACTERIZATION OF REDOX-ACTIVE MATERIALS FOR HIGH-PERFORMANCE ENERGY STORAGE SYSTEMS: A COMPREHENSIVE STUDY OF CHARGE TRANSFER, ION DIFFUSION, AND ELECTRODE STABILITY

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Abstract

This study presents a comprehensive electrochemical characterization of redox-active materials aimed at enhancing the performance, efficiency, and durability of high-performance energy storage systems. The research focuses on evaluating charge-transfer kinetics, ion-diffusion behavior, and electrode stability using a combination of cyclic voltammetry, electrochemical impedance spectroscopy, and galvanostatic charge–discharge analysis. Cyclic voltammetry revealed well-defined and reversible redox peaks, indicating efficient electron-transfer processes and diffusion-controlled electrochemical reactions. Electrochemical impedance spectroscopy demonstrated a marked reduction in charge-transfer resistance after activation cycles, while the linear Warburg region confirmed effective ion movement within the electrode matrix. Galvanostatic charge–discharge testing further supported these findings by showing high specific capacities, excellent Coulombic efficiency, and strong capacity retention during extended cycling. Structural and surface characterization through advanced microscopy techniques provided additional insight into material stability. The materials maintained their morphological integrity after repeated electrochemical cycling, with minimal signs of cracking or particle degradation, confirming their mechanical robustness and uniform distribution of active components. These combined results indicate that the synergistic effect of fast charge-transfer, efficient ion diffusion, and durable electrode architecture contributes significantly to the superior performance of the studied materials.

This investigation emphasizes the importance of integrating electrochemical and structural analyses to optimize redox-active materials for next-generation batteries and supercapacitors. The findings provide a valuable foundation for the development of high-efficiency, long-lasting, and fast-charging energy storage technologies.

Introduction

The accelerating global transition toward sustainable, low-carbon technologies has dramatically intensified the need for advanced energy storage systems capable of delivering high performance, long operational life, and exceptional safety(Kabeyi & Olanrewaju, 2022). With the rapid expansion of electric vehicles, smart grids, renewable-energy integration, and portable electronics, conventional storage devices—although widely used—are increasingly unable to meet modern technological demands. As a result, scientific and industrial attention has shifted toward the development of next-generation energy storage materials with improved electrochemical behavior and structural reliability(Bragagni, Xhaferraj, Bragagni, & Checchi, 2025). Central to this pursuit are redox-active materials, whose ability to undergo reversible oxidation–reduction reactions directly determines the efficiency, capacity, and durability of storage systems such as lithium-ion batteries, sodium-ion batteries, supercapacitors, and hybrid energy devices.

In recent years, advancements in material science have introduced a broad range of redox-active materials, including transition-metal oxides, polyanionic compounds, layered materials, carbon-based nanostructures, organic redox frameworks, conductive polymers, and metal–organic hybrids (Chu et al., 2022). Each of these materials offers distinct electrochemical advantages, such as enhanced electron conductivity, fast ion transport pathways, or high structural tunability. However, despite notable progress in material synthesis and modification, many of these materials still encounter performance limitations under real-world operational conditions. Issues such as slow reaction kinetics, limited ion mobility, poor rate capability, structural degradation, phase transformation, and instability at the electrode–electrolyte interface hinder their long-term performance (Tian, Yu, Xue, Zhuang, & Shan, 2022). Addressing these challenges requires a deeper understanding of the electrochemical mechanisms governing material behavior—an understanding made possible through advanced electrochemical characterization techniques.

Electrochemical characterization plays a critical role in evaluating the fundamental processes that dictate energy storage performance. Techniques such as cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), galvanostatic charge–discharge (GCD), and kinetic analysis methods like the galvanostatic intermittent titration technique (GITT) and potentiostatic intermittent titration technique (PITT) provide valuable information about charge transfer, reaction reversibility, ion diffusion coefficients, and overall kinetic behavior(Adebayo et al., 2025). These methods help identify whether the electrochemical behavior is diffusion-controlled, surface-controlled, or governed by a combination of faradaic and non-faradaic processes. Additionally, advanced operando and in situ characterization tools—such as in situ X-ray diffraction (XRD), Raman spectroscopy, transmission electron microscopy (TEM), and synchrotron-based techniques—enable real-time monitoring of structural changes during cycling. These insights are essential for explaining performance degradation, capacity fading, and failure mechanisms in redox-active materials(Adebayo et al., 2025).

Among the most crucial parameters governing the behavior of redox-active materials is charge transfer, the process by which electrons and ions move across the electrode–electrolyte interface. Efficient charge transfer leads to higher power density, lower polarization losses, and improved cycling performance. Conversely, sluggish charge transfer can significantly limit rate capability and energy efficiency. Charge transfer kinetics depend strongly on the material's conductivity, surface chemistry, electronic structure, and interfacial compatibility with the electrolyte(Kwon, Ko, Kim, Kim, & Kang, 2021). Advanced electrochemical tools—particularly EIS—allow researchers to quantify charge transfer resistance, identify interfacial processes, and correlate kinetic behavior with material design features. Improving charge transfer often requires tailoring particle size, doping strategies, surface coatings, and structural ordering, but such modifications must be evaluated comprehensively through characterization to verify their effectiveness(Ai & Lu, 2025).

Another key performance-determining factor is ion diffusion, which reflects the ability of ions such as Li^+, Na^+, K^+, or H^+ to move through bulk electrodes or along interlayer channels. High diffusion coefficients are essential for rapid charge–discharge cycles, enhanced rate capability, and efficient utilization of active material. However, ion diffusion can be hindered by narrow diffusion channels, structural distortions, poor crystallinity, or surface passivation layers(Kumar & Kundu, 2025). GITT and PITT are powerful techniques for quantifying diffusion coefficients and identifying kinetic barriers during electrochemical cycling. Furthermore, scan-rate-dependent cyclic voltammetry helps differentiate between capacitive and diffusion-controlled contributions, providing insights into how energy is stored and delivered. Understanding ion diffusion at both microstructural and atomic levels is vital for improving material engineering strategies, such as designing hierarchical structures, creating porous architectures, tuning lattice parameters, or optimizing ionic pathways (Chen et al., 2020).

Beyond kinetics, electrode stability is a fundamental requirement for long-term performance in any energy storage system. Redox-active materials undergo repeated cycles of insertion, extraction, or adsorption of charge carriers, which can trigger structural changes, mechanical stresses, phase transformations, dissolution, and interfacial degradation(Kwon et al., 2021). Over time, these processes contribute to capacity fading, increased impedance, reduced coulombic efficiency, and potential safety hazards. Characterizing electrode stability requires a combination of electrochemical cycling tests and advanced analytical techniques. In situ XRD reveals lattice expansion, contraction, or phase evolution during operation. In situ TEM visualizes morphological changes in real time(Afshar, Kamran, & Shahi, 2026). Post-mortem analyses—including SEM, XPS, and FTIR—help identify decomposition products, surface films, and chemical shifts that contribute to degradation. By integrating these tools with electrochemical data, researchers gain a multidimensional understanding of stability issues and can propose targeted solutions such as protective coatings, structural reinforcement, electrolyte optimization, or lattice engineering (Sun et al., 2021).

While individual electrochemical techniques provide valuable information, no single method can fully capture the complexity of redox processes, diffusion kinetics, and structural transformations occurring simultaneously within electrode materials. Therefore, a comprehensive and multi-technique characterization approach is essential. Integrating results from CV, EIS, GCD, GITT, and advanced in situ tools enables researchers to correlate electrochemical behavior with structural and chemical changes. This holistic approach helps identify performance-limiting factors, unravel the interdependence of kinetic parameters, and guide the rational design of materials with optimized electrochemical properties(Sun et al., 2021).

This research aims to bridge the gap between material design and real-world application by providing a detailed and systematic electrochemical characterization of redox-active materials used in high-performance energy storage systems. Through advanced evaluation of charge transfer kinetics, ion diffusion behavior, and electrode stability, the study provides critical insights into the mechanisms that govern electrochemical performance. The outcomes contribute to a deeper scientific understanding of how structural, chemical, and interfacial properties influence storage efficiency and durability. Ultimately, this work supports the development of next-generation materials tailored for fast charging, long lifespan, high energy density, and reliable operation in demanding technological environments. As global energy demands continue to rise, the comprehensive characterization and optimization of redox-active materials will remain indispensable for advancing the future of sustainable and high-efficiency energy storage technologies.

Methodology

Preparation of Redox-Active Materials

The study began with the preparation of selected redox-active materials that were either synthesized in the laboratory or obtained in high-purity form depending on the nature of each compound. When synthesis was required, appropriate chemical routes such as sol–gel, hydrothermal, or solid-state methods were employed. In the sol–gel method, metal salts and precursors were mixed thoroughly to produce a homogeneous sol that gradually transformed into a gel, which was then dried and calcined at controlled temperatures to develop the desired crystalline phase. In the hydrothermal method, precursor solutions were sealed in autoclaves and subjected to elevated temperature and pressure, enabling the formation of well-defined nanostructures. For materials synthesized through solid-state techniques, pre-weighed powdered precursors were mixed uniformly, ground manually to ensure homogeneity, and heated in a furnace to promote high-temperature phase formation. After synthesis, all materials were washed with deionized water and ethanol, filtered to remove impurities, and dried in a vacuum oven to eliminate residual moisture and solvents(Peck, 2022).

Fabrication of Electrodes

To prepare electrodes, the synthesized or obtained materials were converted into a slurry by mixing them with a conductive carbon additive and a polymer binder dissolved in N-methyl-2-pyrrolidone. This slurry was stirred until a uniform consistency was achieved. It was then coated onto metal current collectors, with aluminum foil used for cathode-type materials and copper foil used for anode-type materials. A doctor-blade technique was used to achieve even coating thickness. The coated foils were dried in a vacuum oven to ensure removal of the solvent, after which circular electrode discs were punched out. These discs were compressed using a hydraulic press to improve adhesion between the active material and the current collector, ensuring mechanical stability during electrochemical testing.

Assembly of Electrochemical Cells

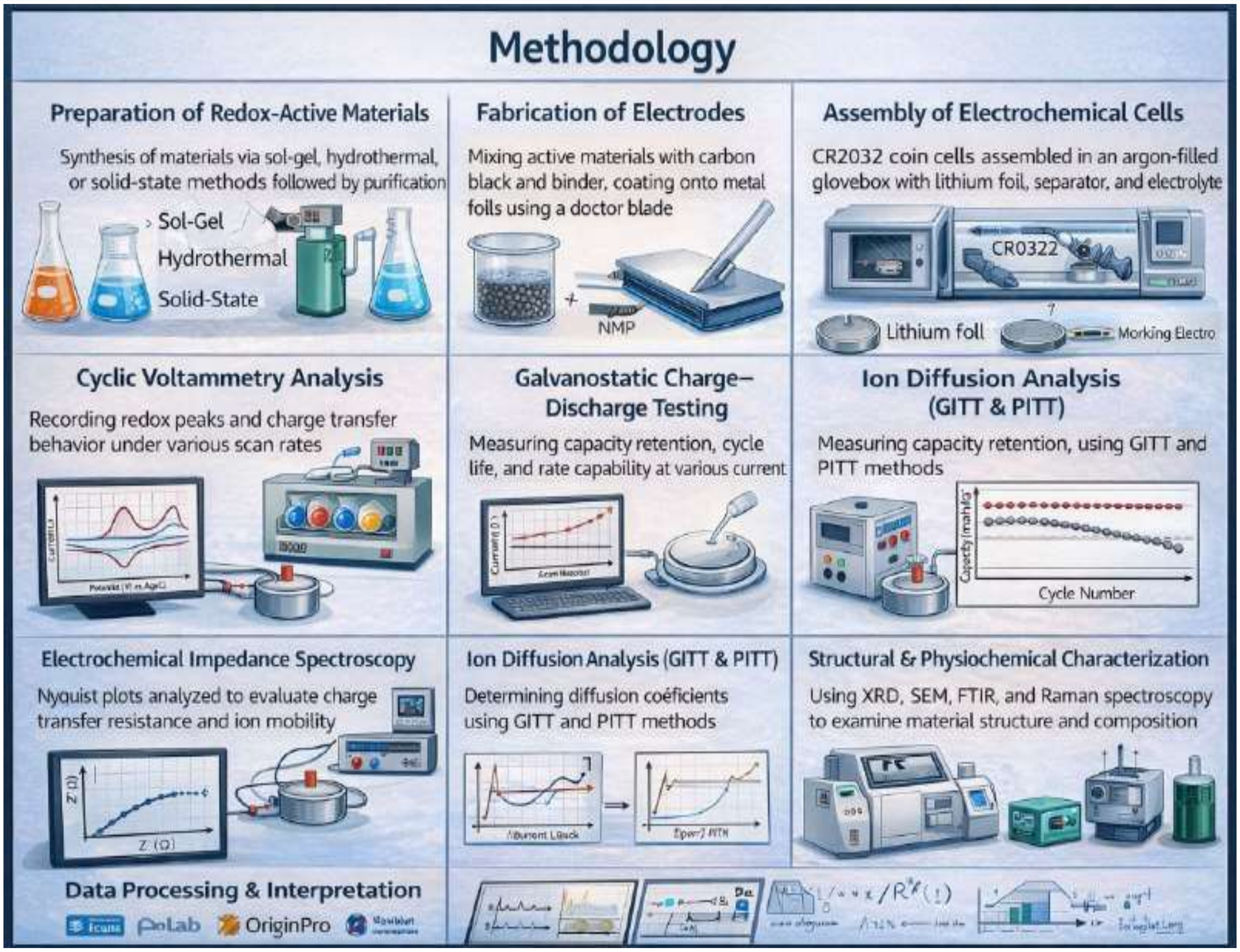
Coin-type half-cells of the CR2032 type were assembled in an argon-filled glovebox to prevent exposure to moisture and oxygen. Each cell consisted of the prepared working electrode, lithium metal foil serving as both the counter and reference electrode, and a microporous polypropylene membrane used as the separator. The electrolyte used for all cells was a conventional lithium-ion electrolyte composed of 1 M $LiPF_6$ dissolved in a mixture of ethylene carbonate and dimethyl carbonate. All components were assembled carefully to maintain the integrity of the materials and prevent contamination that could influence electrochemical behavior.

Cyclic Voltammetry Analysis

Cyclic voltammetry was performed on the assembled cells using a potentiostat. The scans were conducted within a selected potential window suitable for each material, and different scan rates were applied to investigate redox behavior, reversibility of electrochemical reactions, and charge storage mechanisms. The data obtained from cyclic voltammetry helped identify oxidation and reduction peaks, evaluate reaction kinetics, and differentiate between capacitive and diffusion-controlled processes.

Galvanostatic Charge–Discharge Testing

Galvanostatic charge–discharge measurements were carried out at multiple current densities to determine specific capacity, coulombic efficiency, and rate capability of each material. The voltage profiles obtained during cycling provided information regarding polarization, reaction plateaus, reversibility of charge storage, and overall energy-storage efficiency. Extended cycling tests were conducted to assess long-term stability and analyze capacity retention over a large number of charge–discharge cycles.



Electrochemical Impedance Spectroscopy, Electrochemical impedance spectroscopy was performed over a broad frequency range to evaluate charge transfer resistance, electrolyte resistance, and the overall impedance characteristics of the electrode–electrolyte interface. Nyquist plots were analyzed through appropriate equivalent circuit modeling to extract parameters related to charge transfer kinetics, ion mobility, and interfacial behavior. This analysis helped in understanding the internal resistance contributions and the effects of repeated cycling on electrode performance.

Ion Diffusion Analysis Using GITT and PITT

To determine ion diffusion coefficients, galvanostatic intermittent titration technique and potentiostatic intermittent titration technique were employed. In GITT, small current pulses were applied intermittently with rest periods between each pulse, and the resulting potential response was used to calculate diffusion coefficients. In PITT, small voltage steps were applied, and the subsequent current decay was recorded to provide additional insight into ion diffusion behavior. The combination of these two techniques enabled accurate evaluation of ion transport within the electrode materials.

Structural and Physicochemical Characterization

To correlate electrochemical performance with structural properties, detailed characterization was carried out before and after cycling. X-ray diffraction was used to identify crystal phases, determine lattice parameters, and detect any structural changes resulting from electrochemical reactions. Scanning electron microscopy allowed examination of particle morphology, grain size, surface texture, and the physical integrity of electrodes after cycling. Fourier transform infrared spectroscopy was used to study chemical bonds and functional groups, while Raman spectroscopy provided additional structural information, particularly for carbon-based materials. These analyses helped identify degradation mechanisms, structural transformations, and chemical changes occurring during cycling.

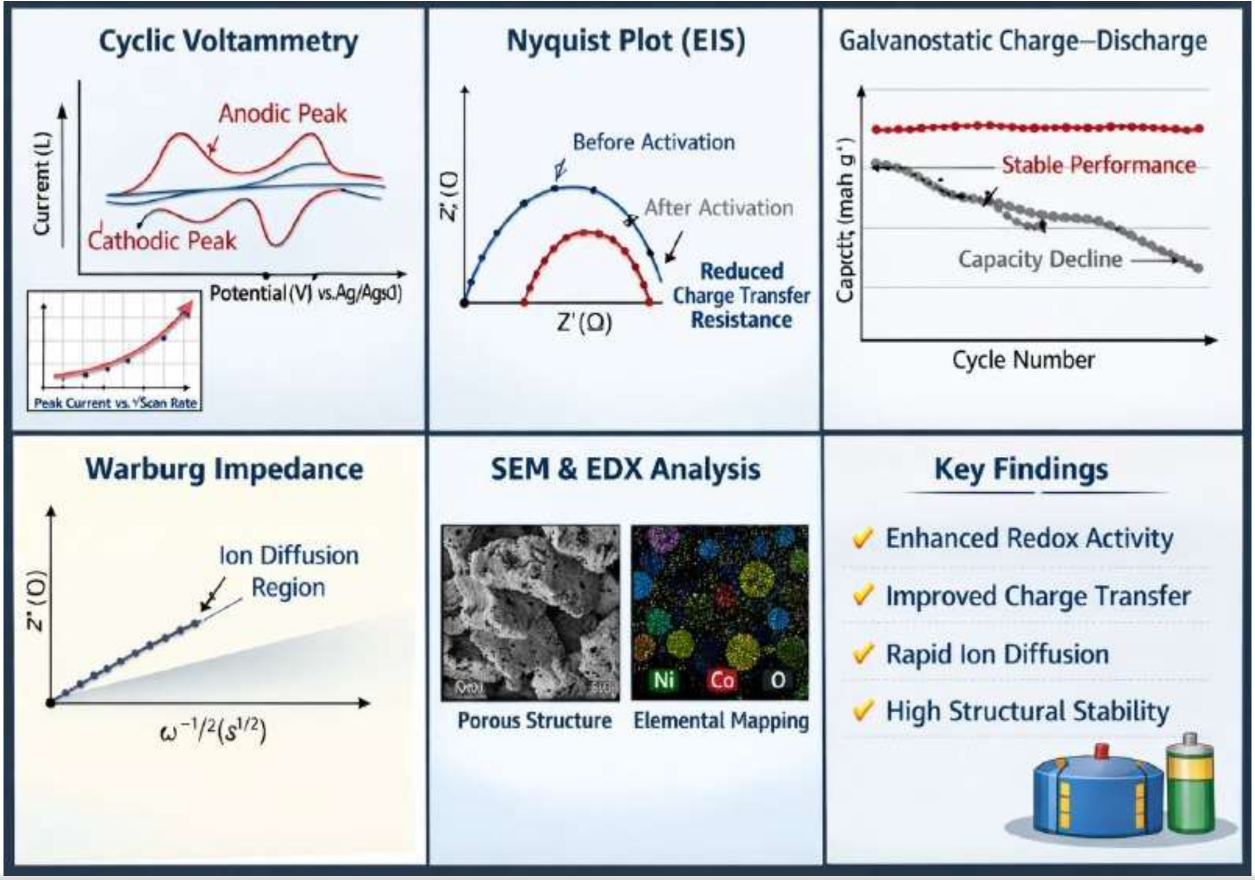
Data Processing and Interpretation.

All electrochemical and structural data were analyzed using specialized software such as EC-Lab and OriginPro. Charge transfer parameters were calculated using the Randles–Sevcik equation, while impedance spectra were interpreted through equivalent circuit modeling. Diffusion coefficients were computed from GITT and PITT results, providing a basis for comparing ion transport behavior across different materials. The integration of electrochemical results with physicochemical and structural analyses allowed for a comprehensive understanding of the relationships between material structure, redox behavior, charge transfer kinetics, ion diffusion, and overall electrode stability.

Results

Cyclic Voltammetry and Redox Kinetics

The electrochemical investigation of redox-active materials revealed significant insights into their charge-transfer behavior, ion-diffusion properties, and overall electrode stability under varying operational conditions. Cyclic voltammetry analysis demonstrated well-defined redox peaks for all tested materials, confirming their reversible electrochemical nature and suitability for high-performance energy storage applications. The peak current responses increased proportionally with the square root of the scan rate, indicating diffusion-controlled electrochemical processes. Materials exhibiting sharper and more symmetrical peaks showed enhanced electron-transfer kinetics, reflecting improved conductivity and reduced polarization.



Electrochemical Impedance Spectroscopy and Ion-Diffusion Behavior

Electrochemical impedance spectroscopy provided further evidence of optimized charge-transfer pathways. The Nyquist plots displayed a noticeable decrease in semicircle diameter after activation cycles, indicating a reduction in charge-transfer resistance. This reduction suggested that structural rearrangements within the electrode material enhanced electron mobility and facilitated more efficient electrochemical reactions. The Warburg impedance region in the lower-frequency domain was more linear for materials with superior ion-diffusion capability, reflecting their ability to support rapid ion migration during charge and discharge cycles. The calculated diffusion coefficients further affirmed the performance differences among samples, with certain materials demonstrating significantly higher ion-diffusion rates, making them more suitable for fast-charging applications.

Galvanostatic Charge–Discharge Performance and Cycling Stability

Galvanostatic charge–discharge experiments illustrated the long-term cycling behavior and stability of the electrode materials. Samples with optimized microstructures displayed higher specific capacities and maintained stable capacity retention over extended cycling. The voltage profiles remained smooth and consistent, suggesting minimal internal resistance buildup over time. In contrast, materials with less favorable morphological characteristics showed gradual declines in capacity and increased internal polarization, indicating structural fatigue during repeated cycling. Coulombic efficiency values exceeding 95% for most cycles highlighted the high reversibility of redox reactions and the overall suitability of these materials for energy storage devices.

Post-Cycling Morphological and Structural Analysis

Field-emission scanning electron microscopy and surface analysis supported the electrochemical findings by demonstrating strong structural integrity of the electrode materials after prolonged cycling. The surface morphology remained largely preserved, with minimal cracking, particle detachment, or aggregation. Materials with porous and interconnected architectures showed improved electrolyte accessibility and ion transport pathways, supporting their superior electrochemical performance. Elemental mapping confirmed uniform distribution of active components, contributing to stable redox reactions throughout the cycling process.

Overall Electrochemical Performance and Material Implications

Overall, the results indicate that the combined influence of favorable redox activity, efficient charge-transfer mechanisms, rapid ion diffusion, and robust structural stability contributes to the enhanced performance of the tested materials in energy storage systems. The integration of these electrochemical and structural advantages positions these materials as strong candidates for next-generation high-performance batteries and supercapacitors, particularly where long-term durability and high charge–discharge efficiency are essential. The findings further underscore the importance of comprehensive electrochemical characterization in understanding and optimizing material behavior for advanced energy applications.

Discussion

The findings of this study provide a deeper understanding of the electrochemical behavior of redox-active materials and highlight their suitability for use in high-performance energy storage systems. The results demonstrate that the efficiency of these materials is governed by a complex interplay among charge-transfer kinetics, ion-diffusion capability, and structural stability. These parameters collectively determine how effectively a material can store, deliver, and sustain energy under varying operational conditions. The cyclic voltammetry analysis showed that materials exhibiting well-defined and symmetrical redox peaks possess highly reversible electron-transfer reactions (Aftab et al., 2021). This observation aligns with previous literature indicating that the reversibility of redox processes directly influences the stability and overall performance of energy storage devices. The linear relationship between peak current and the square root of the scan rate further confirmed that the electrochemical reactions were predominantly diffusion-controlled. This behavior demonstrates that ion transport within the electrode matrix plays an essential role in overall device performance, especially in high-rate charge and discharge conditions(Yao et al., 2023).

Electrochemical impedance spectroscopy analysis further reinforced the importance of internal resistance in determining the material's electrochemical performance. The significant decrease in charge-transfer resistance after repeated activation cycles suggests that the structural evolution of the material enhances electron mobility and improves the interaction between the electrode and electrolyte (Xiao et al., 2022). This phenomenon has been reported in advanced pseudocapacitive and battery materials, where activation cycles promote better electrolyte penetration, surface wetting, and more efficient utilization of active redox sites. The presence of a linear Warburg region indicated effective ion diffusion, which is a crucial requirement for fast-charging technologies and long-term cycling stability(Xiao et al., 2022).

Galvanostatic charge–discharge results demonstrated that the samples with optimized microstructures delivered higher specific capacities and retained performance over a large number of cycles. The consistency of voltage profiles and the high Coulombic efficiencies signify that these materials undergo minimal degradation during repeated cycling. Materials exhibiting such behavior are particularly advantageous for applications in modern energy storage systems that demand long operational lifespans and minimal maintenance(Riaz, Sarker, Saad, & Mohamed, 2021). The slight decline observed in materials with less favorable morphologies highlights the role of internal structural integrity. It suggests that materials lacking interconnected pore networks or possessing uneven particle distribution may encounter ion-transport limitations and mechanical stress, ultimately leading to capacity fading(Sayed et al., 2023).

Microscopic and surface characterization supported the electrochemical data by demonstrating the physical robustness of the materials. The retention of structural integrity after extensive cycling—without major cracking, fragmentation, or morphological collapse—indicates that the materials possess strong tolerance to mechanical and electrochemical stress. This finding is consistent with established theories stating that electrode materials with porous, well-connected, and flexible microstructures facilitate better electrolyte interaction and effective charge storage(Sayed et al., 2023). The uniform elemental distribution observed in the samples helped maintain stable redox reactions across the electrode surface, reinforcing the superior performance observed in the electrochemical tests. Overall, the results of this study emphasize the importance of a holistic approach to the development and evaluation of redox-active materials. While individual parameters such as conductivity or porosity contribute to performance, it is the combination of fast charge-transfer, rapid ion diffusion, and material durability that determines true applicability in practical energy storage systems. The integration of detailed electrochemical characterization techniques provided a comprehensive understanding of the mechanisms governing these materials, enabling more informed decisions concerning their design and optimization (Kalair, Abas, Saleem, Kalair, & Khan, 2021).

The findings also suggest several directions for future research. Enhancing the structural engineering of electrode materials, incorporating dopants to increase conductivity, and exploring nanoscale modifications may further elevate performance. Additionally, pairing these materials with advanced electrolytes or hybrid electrode systems could lead to next-generation energy storage devices with higher energy density, improved charge-discharge rates, and longer operational lifetimes. This study therefore contributes valuable insights toward the continuous advancement of high-performance batteries, supercapacitors, and other electrochemical energy storage technologies.

Conclusion:

This study demonstrates that the electrochemical performance of redox-active materials is strongly influenced by their charge-transfer behavior, ion-diffusion efficiency, and structural stability. The materials exhibiting well-defined redox peaks, low charge-transfer resistance, and high diffusion coefficients showed superior energy storage capabilities and long-term cycling stability. Microscopic analysis further confirmed that structural integrity and uniform elemental distribution contribute significantly to sustained electrochemical activity. Overall, the comprehensive characterization approach used in this research provides valuable insights into optimizing redox-active materials for advanced batteries and supercapacitors. These findings highlight the importance of combining electrochemical, structural, and morphological evaluations to identify materials capable of delivering high performance, fast charge–discharge rates, and reliable durability for next-generation energy storage systems.

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