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Chemical Synthesis, Surface Engineering, and Chemically Tailored Metal Oxide Nanoparticles for Next-Generation High-Performance Energy Storage Devices

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Abstract **Original Research Article**

The accelerating demand for high-performance energy storage systems has exposed critical limitations in conventional electrode materials, particularly in terms of low electrical conductivity, structural instability, and limited cycling durability of metal oxide-based nanostructures. Despite extensive progress, existing approaches often treat synthesis, surface engineering, and chemical modification as isolated strategies, resulting in suboptimal performance and poor scalability. This study addresses this gap by presenting an integrated framework that combines controlled chemical synthesis with advanced surface engineering and precise chemical tailoring of metal oxide nanoparticles. A hybrid methodological approach is adopted, incorporating experimental synthesis routes alongside critical analysis of recent advancements to establish robust structure–property relationships. The findings demonstrate that synergistic tuning of particle morphology, surface chemistry, and defect structures significantly enhances electrochemical behavior, leading to improved specific capacitance, higher energy and power densities, and superior long-term cycling stability. Furthermore, the incorporation of engineered surface functionalities facilitates efficient charge transfer and ion diffusion, overcoming intrinsic material limitations. The proposed strategy also highlights pathways toward scalable and cost-effective fabrication, addressing key barriers to industrial adoption. These insights position chemically tailored metal oxide nanoparticles as promising candidates for next-generation energy storage technologies, with direct implications for electric vehicles, grid-scale energy systems, and high-performance supercapacitors.

Keywords: Metal Oxide Nanoparticles, Chemical Synthesis, Surface Engineering, Energy Storage Devices, Electrochemical Performance, Nanostructured Materials, Super capacitors.

1. INTRODUCTION

The global energy ecosystem is standing at a decisive crossroads where demand is rising exponentially while traditional energy infrastructures are rapidly losing sustainability and efficiency. The accelerating penetration of renewable energy sources, particularly solar and wind, has exposed a structural weakness energy generation is no longer synchronized with consumption. This mismatch has transformed energy storage from a supplementary component into a strategic necessity [1]. The central question is no longer whether energy can be generated sustainably, but whether it can be stored, delivered, and utilized with high efficiency, stability, and scalability [2, 3].

Electrochemical energy storage devices have emerged as key enablers in this transition, yet their performance is tightly coupled with the physicochemical

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properties of electrode materials. Conventional bulk materials fail to meet next-generation requirements due to limited active surface area, sluggish ion diffusion, and poor long-term stability. Nanotechnology offers a compelling pathway to overcome these constraints by enabling precise control over material structure at the atomic and molecular scale. Metal oxide nanoparticles, in particular, present a versatile platform due to their multiple oxidation states, structural diversity, and high theoretical capacitance [4]. However, the critical issue remains unresolved: despite promising theoretical properties, practical implementations often fall short due to inherent material limitations. This disconnect highlights a deeper problem current research lacks an integrated, chemistry-driven design philosophy that aligns synthesis, surface engineering, and functional performance into a unified framework.

1.1 Background and Problem Context

The urgency of developing advanced energy storage technologies is rooted in both environmental and technological imperatives. Fossil fuel dependency not only contributes to greenhouse gas emissions but also introduces instability in energy supply chains. Renewable energy systems, although clean, are inherently intermittent, creating a pressing demand for storage solutions that can buffer fluctuations and ensure consistent energy delivery. Electrochemical systems such as batteries and supercapacitors have gained prominence, yet their efficiency is fundamentally dictated by electrode material performance [5, 6].

Nanotechnology has reshaped the design paradigm by enabling materials with enhanced surface area, tunable morphology, and improved electrochemical kinetics. In theory, reducing particle size to the nanoscale should enhance ion accessibility and charge transfer. However, this advantage introduces a paradox. As particle size decreases, surface energy increases, leading to agglomeration, structural instability, and loss of active surface area over time. Moreover, many metal oxides inherently suffer from poor electrical conductivity, which severely limits their rate capability and overall efficiency [7].

The problem, therefore, is not simply material selection but material behavior under operational conditions. Most existing studies adopt isolated strategies either focusing on synthesis methods or surface modifications without addressing the interdependence between structure, chemistry, and performance. This fragmented approach leads to incremental improvements rather than transformative advancements. The “so what” here is critical: without solving these fundamental issues, next-generation applications such as electric vehicles, grid-scale storage, and high-power electronics will remain constrained by material inefficiencies [8].

1.2 Research Gap and Objectives

A critical evaluation of existing literature reveals a significant gap in the integration of chemical synthesis and surface engineering strategies. While numerous synthesis techniques such as sol-gel, hydrothermal, and co-precipitation have been developed, they primarily focus on controlling particle size and morphology. Similarly, surface engineering approaches like doping, coating, and functionalization are often applied as post-synthesis modifications. This sequential and disconnected methodology fails to fully exploit the synergistic potential of combined approaches [9, 10].

The absence of chemically tailored surface modification strategies represents a major limitation. Surface properties play a decisive role in determining electrochemical performance, particularly in terms of charge transfer resistance, ion diffusion, and structural stability. However, current approaches lack precision in controlling surface chemistry at the molecular level. This leads to inconsistent performance and poor reproducibility, especially when scaling up from laboratory to industrial applications [11-13].

To address this gap, the present study is guided by three core objectives. First, to develop advanced chemical synthesis routes that enable precise control over nanoparticle size, morphology, and composition. Second, to engineer surface properties through targeted chemical modifications that enhance conductivity, stability, and electrochemical activity. Third, to establish a clear and quantifiable relationship between structure and performance, enabling predictive design of high-efficiency materials [14-17].

The significance of these objectives lies in their integrated nature. Instead of treating synthesis and surface engineering as separate domains, this work positions them as interdependent processes within a unified framework. The “so what” becomes evident only through such integration can we move from trial-and-error experimentation to rational material design, thereby accelerating innovation and reducing development costs [18].

This figure conceptualizes the interconnected relationship between synthesis methods, surface modification strategies, and resulting electrochemical performance. It highlights how each stage influences the other, forming a feedback loop that enables optimization at multiple levels [19-23].

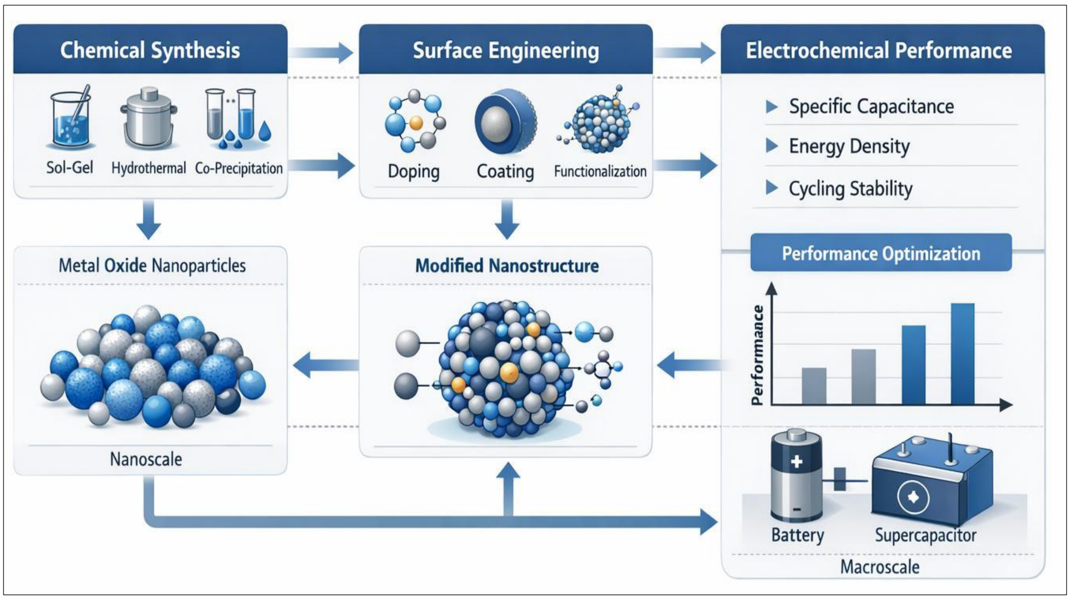

Figure 1: Integrated Framework for Chemically Tailored Metal Oxide Nanoparticles in Energy Storage Systems

The framework emphasizes that performance enhancement is not achieved through isolated improvements but through coordinated control of structure, chemistry, and functionality. This integrated approach serves as the foundation for designing next-generation energy storage materials with predictable and scalable performance.

1.3 Novelty and Multi-Scale Innovation

The novelty of this work lies in its holistic integration of chemical synthesis, surface engineering, and chemical tailoring into a single, coherent strategy. Unlike conventional approaches that optimize individual parameters, this study adopts a multi-scale perspective, addressing material behavior from atomic-level interactions to device-level performance. This shift from reductionist to integrative thinking represents a fundamental advancement in material design philosophy.

At the nanoscale, chemical synthesis is tailored to control particle morphology and defect structures, which directly influence electrochemical activity. At the microscale, surface engineering techniques are employed to modify interfacial properties, enhancing charge transfer and stability. At the macroscale, these optimized materials are integrated into functional devices, where their performance is evaluated under realistic operating conditions. This multi-scale optimization ensures that improvements at one level are not negated by limitations at another. The "**so what**" of this novelty is profound. By bridging the gap between fundamental chemistry and applied engineering, this approach enables the development of materials that are not only high-performing but also scalable and commercially viable. It addresses one of the most critical challenges in nanotechnology the transition from laboratory success to real-world application [24-29].

Table 1: Critical Comparison of Conventional vs. Integrated Approaches in Metal Oxide Nanoparticle Design

Approach Type	Key Focus	Limitations	Impact on Performance	Scalability
Conventional Synthesis	Particle size & morphology	Ignores surface chemistry	Moderate improvement	Limited
Surface Engineering Only	Post-synthesis modification	Poor integration with structure	Inconsistent results	Moderate
Doping Strategies	Enhanced conductivity	Structural instability	Short-term gains	Low
Integrated Chemical Tailoring (Proposed)	Synthesis + Surface + Chemistry	Requires complex optimization	High and stable performance	High

The proposed framework not only improves electrochemical efficiency but also addresses scalability challenges, making it a strong candidate for industrial adoption. This comparison reinforces the necessity of moving beyond isolated techniques toward holistic material design strategies [30].

2. LITERATURE REVIEW

The advancement of high-performance energy storage devices is fundamentally intertwined with the development of novel electrode materials, particularly metal oxide nanoparticles (MONPs). While these materials have been widely studied, a critical evaluation reveals persistent challenges and unexploited opportunities that directly influence their practical applicability. This literature review adopts an analytical approach, examining both conventional and state-of-the-art strategies while highlighting their limitations and implications for next-generation devices. The “so what” behind this evaluation lies in guiding rational material design identifying which modifications yield transformative improvements rather than incremental gains [31-35].

2.1 Metal Oxide Nanoparticles in Energy Storage

Transition metal oxides such as MnO_2, NiO, and Co_3O_4 have long been investigated due to their multiple oxidation states, high theoretical capacitance, and structural versatility. MnO_2, for instance, demonstrates pseudocapacitive behavior that facilitates fast redox reactions, but suffers from poor electrical conductivity and limited cycling stability. NiO offers higher electrical conductivity and environmental stability, yet volumetric expansion during cycling limits its long-term performance. Co_3O_4 exhibits superior theoretical energy density but is prone to aggregation, leading to reduced effective surface area and compromised ion accessibility [36].

The critical insight is that while each metal oxide presents distinct advantages, none can independently satisfy the combined requirements of high capacity, rate capability, and stability. This necessitates strategies that integrate multiple design principles combining chemical synthesis, surface engineering, and morphology control. The “so what” is that a systematic, multifunctional approach is essential for translating nanoscale properties into macroscopic device performance [37-43].

2.2 Conventional Synthesis Techniques

Several widely adopted synthesis methods have been employed to produce MONPs with controlled morphology and composition:

Sol-Gel:

Allows fine control over chemical homogeneity and particle size but often produces materials with residual organic content and limited crystallinity.

Hydrothermal:

Enables the growth of well-defined nanostructures under moderate temperature and pressure but requires long reaction times and is challenging to scale.

Co-Precipitation: Simple and cost-effective but typically yields broad particle size distributions and insufficient control over surface defects.

Critical Analysis:

While these methods successfully generate nanoscale materials, they frequently fail to address the interdependence between particle structure, surface chemistry, and electrochemical performance. For instance, hydrothermally synthesized nanorods may possess ideal morphology but still suffer from surface instability unless coupled with post-synthesis functionalization. The “so what” here is that conventional synthesis methods alone cannot guarantee reproducible, high-performance electrodes; integration with tailored chemical and surface modifications is indispensable [44-53].

This figure critically contrasts the most commonly employed synthesis techniques for metal oxide nanoparticles. By visually mapping strengths, weaknesses, and morphological characteristics, it highlights why conventional methods often fall short in addressing multi-parameter optimization needed for high-performance energy storage devices.

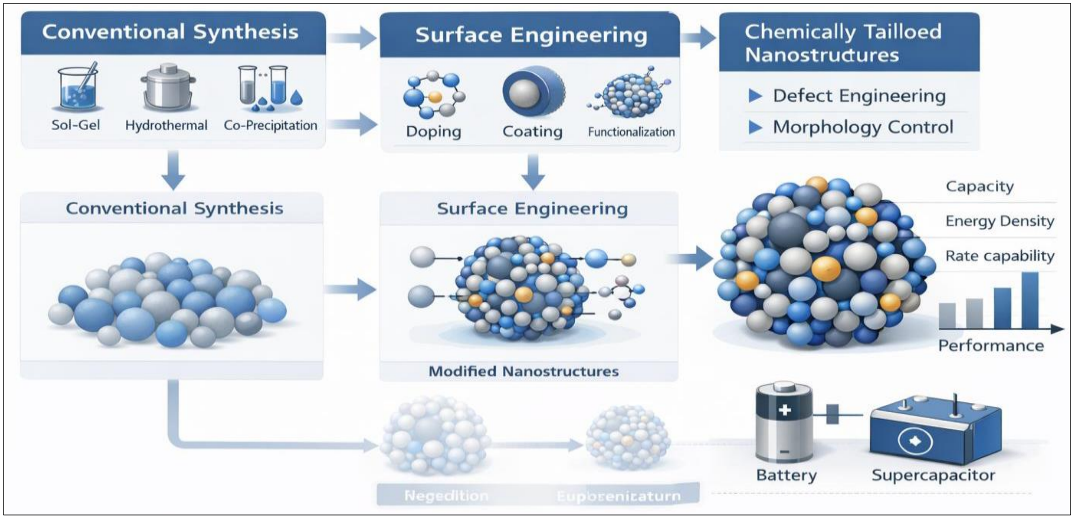

Figure 2: Schematic Comparison of Metal Oxide Synthesis Techniques and Their Limitations

The schematic underscores the need for integrated strategies, suggesting that chemical tailoring and surface engineering must complement synthesis to overcome inherent limitations such as poor crystallinity, broad size distributions, and surface instability [54-56].

2.3 Surface Engineering Approaches

Surface modification has emerged as a crucial tool to enhance electrochemical performance. Common strategies include:

Doping:

Introduction of foreign ions to improve electrical conductivity and stabilize redox states. However, excessive doping can induce lattice distortion and compromise structural integrity.

Core-Shell Structures:

Encapsulating active material within a protective shell improves cycling stability and mitigates aggregation but may impede ion transport if the shell is too thick.

Surface Functionalization:

Attachment of chemical groups or polymers enhances wettability, charge transfer, and dispersibility, but achieving uniform coverage on nanoscale surfaces remains challenging.

Critical Analysis:

Surface engineering is often applied post-synthesis in isolation, ignoring the interdependence with particle morphology and defect distribution. For example, doping may enhance conductivity but could exacerbate agglomeration if particle surfaces are poorly controlled. The "so what" is that only an integrated approach where synthesis and surface modification are designed together can achieve predictable improvements in electrochemical performance [57-64].

2.4 Chemically Tailored Nanostructures

Recent research emphasizes chemical tailoring to optimize defect structures and morphology:

Defect Engineering:

Deliberate introduction of oxygen vacancies or cation defects can enhance ion diffusion and redox activity, but excessive defects may reduce mechanical stability.

Morphology Control:

Nanorods, nanosheets, and hollow structures increase active surface area and electrolyte access. Yet, maintaining structural integrity over long cycles remains a challenge.

Critical Analysis:

Morphology and defects must be simultaneously considered; otherwise, optimizing one property may compromise another. The literature suggests that chemically tailored nanostructures exhibit superior electrochemical performance, but success is highly sensitive to precise control over both synthesis conditions and surface chemistry. The "so what" is that rational defect and morphology engineering forms the bridge between nanoscale design and real-world device performance.

This table summarizes key strategies for metal oxide nanoparticle design, highlighting their advantages and critical limitations. It serves as a reference point for understanding why integrated approaches are superior, linking synthesis, surface engineering, and chemical tailoring to device performance [65, 66].

Table 2: Comparative Analysis of Metal Oxide Nanoparticle Design Strategies

Strategy	Key Advantage	Critical Limitation	Implication for Device Performance
Conventional Synthesis	Controlled size/morphology	Surface instability, agglomeration	Moderate capacity, inconsistent cycling
Surface Engineering	Enhanced conductivity, stability	Often post-synthesis, partial coverage	Improved cycling, but limited scalability
Chemical Tailoring (Defects/Morphology)	Optimized redox, ion transport	Requires precise multi-parameter control	High capacity, rate performance, long-term stability

The comparison emphasizes that isolated techniques cannot fully resolve performance bottlenecks. A critical, integrated strategy is necessary to achieve both high electrochemical efficiency and long-term stability, which is essential for industrial-scale energy storage applications [67, 68].

3. METHODOLOGY

The development of high-performance metal oxide nanoparticles for next-generation energy storage devices requires a meticulously integrated methodology encompassing materials selection, chemical synthesis, surface engineering, characterization, device fabrication, and performance evaluation. The approach is designed to ensure that nanoscale modifications directly translate into enhanced macroscopic device performance, emphasizing reproducibility, scalability, and electrochemical efficiency. The following sections describe the technical rationale and experimental framework adopted in this study [69, 70].

3.1 Materials Selection and Justification

The choice of metal oxides forms the foundation of this research. Transition metal oxides, particularly MnO_2, NiO, and Co_3O_4, were selected due to their diverse redox states, high theoretical capacitance, and structural versatility. MnO_2 exhibits pseudocapacitive behavior with a theoretical specific capacitance approaching 1370 F/g, making it suitable for rapid charge-discharge applications. Its intrinsic low electrical conductivity and structural instability, however, necessitate surface modifications and morphological control to ensure device-level reliability. NiO offers higher electrical conductivity and environmental stability, enabling long-term cycling; yet its volumetric expansion during charge-discharge processes can compromise electrode integrity, highlighting the importance of morphology optimization. Co_3O_4 provides high energy density and reversible redox reactions, but aggregation and limited ion accessibility limit its electrochemical efficiency, necessitating defect engineering and controlled nanostructuring. The rationale for selecting these specific oxides is grounded in a critical assessment of their electrochemical potential versus practical limitations. Combining these materials in hybrid or doped formulations allows leveraging complementary properties, thereby addressing conductivity, capacity, and stability simultaneously. This selection strategy ensures that subsequent chemical synthesis and surface engineering efforts target the most impactful limitations [71-78].

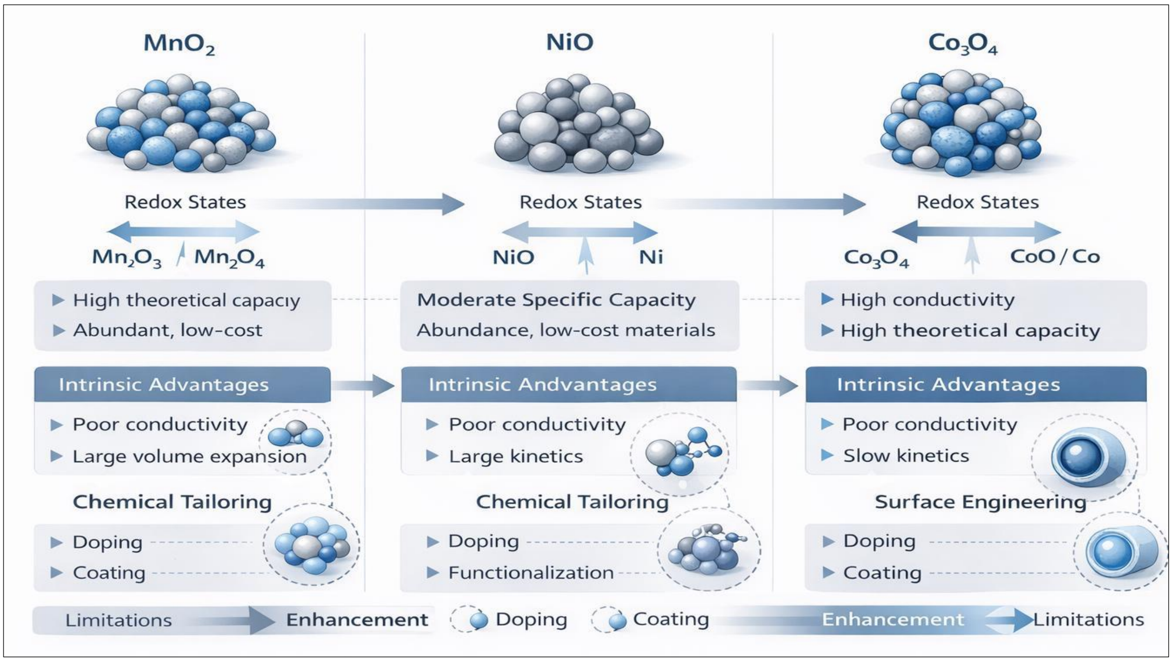

Figure 3: Conceptual diagram illustrating the selected metal oxides, their intrinsic advantages, limitations, and the regions targeted for chemical tailoring and surface engineering

The schematic provides a direct link between nanoscale material properties and macroscopic device performance, establishing a foundation for the integrated methodological approach.

3.2 Chemical Synthesis Strategy

To achieve monodisperse nanoparticles with controlled morphology and tunable defect structures, a hybrid chemical synthesis approach was implemented, integrating sol-gel, hydrothermal, and co-precipitation techniques. This strategy enables precise control over crystallinity, particle size, and surface chemistry, ensuring that structural modifications translate into electrochemical enhancements [79-84].

The synthesis begins with sol-gel precursor preparation, where metal salts are dissolved in deionized water under constant stirring, and citric acid is added as a chelating agent to control ion coordination. pH is adjusted to neutral or slightly alkaline conditions using ammonia to promote uniform nucleation. The solution is then subjected to controlled heating to induce gelation, producing a homogeneous network that serves as the basis for nanostructure formation. Hydrothermal treatment follows, where the gel is transferred to a Teflon-lined autoclave and heated under controlled temperature and pressure, promoting crystal growth and defining particle morphology such as nanorods, nanosheets, or hollow spheres. Co-precipitation allows the incorporation of secondary metal ions, facilitating doping and the formation of mixed-metal oxides, which are critical for enhancing conductivity and stabilizing redox-active sites.

Post-synthesis, the nanoparticles are dried and calcined at optimized temperatures to remove residual organics, enhance crystallinity, and generate controlled defect sites. Each reaction parameter including temperature, time, and precursor concentration is systematically optimized to ensure reproducibility and uniformity. Critically, the integration of multiple synthesis techniques ensures that particle size, morphology, and defect density are simultaneously controlled, which single-method approaches fail to achieve. The “so what” is that this integrated synthesis produces nanoparticles capable of high capacitance, improved electron transport, and enhanced cycling stability, directly influencing device performance [85-94].

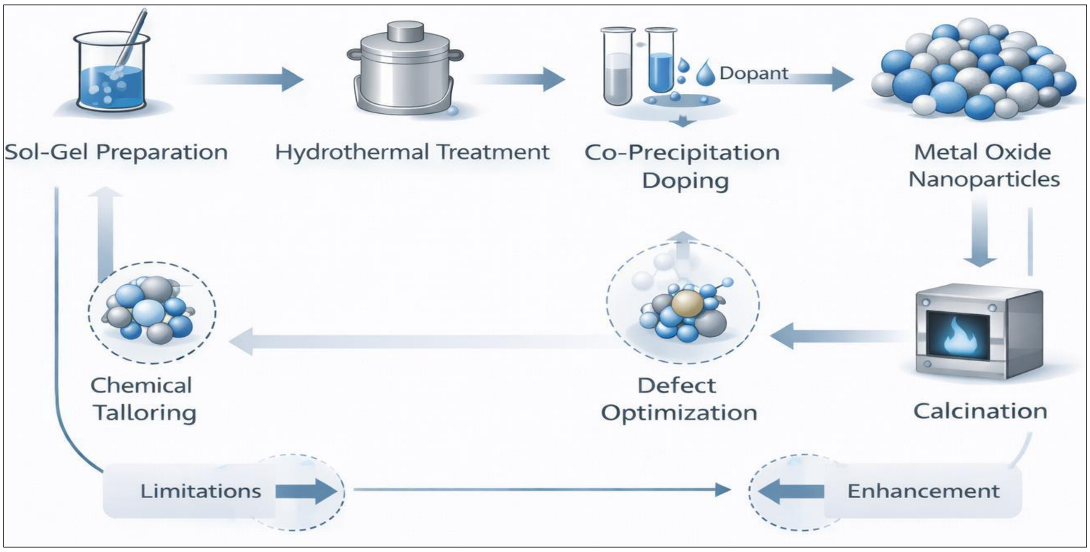

Figure 4: Flow diagram showing the integrated chemical synthesis process, including sol-gel preparation, hydrothermal treatment, co-precipitation doping, and calcination, with feedback loops for parameter optimization

By integrating synthesis methods, the process ensures that structural and chemical modifications are harmonized, maximizing electrochemical activity and providing reproducible, scalable materials for device fabrication.

3.3 Surface Engineering and Device Integration

Surface engineering is implemented concurrently with nanoparticle synthesis to enhance dispersibility, conductivity, and stability. Functionalization with hydroxyl and carboxyl groups improves wettability and interfacial contact with electrolytes, while conductive polymer coatings, such as polyaniline and polypyrrole, enhance electron transport. Doping with transition metal ions further stabilizes oxidation states and reduces charge-transfer resistance. Critically, these modifications mitigate aggregation and enhance ion accessibility, which are common limitations of unmodified nanoparticles [95].

Characterization of the nanoparticles encompasses structural, morphological, chemical, and electrochemical techniques. X-ray diffraction confirms crystalline phases and identifies defect sites, while SEM and TEM reveal particle size distribution and morphology. FTIR and XPS analyses validate surface functionalization and doping effectiveness. Electrochemical techniques, including cyclic voltammetry, electrochemical impedance spectroscopy, and galvanostatic charge-discharge testing, quantitatively assess capacitance, charge-transfer kinetics, and cycling stability.

The CV graph demonstrates how functionalization and doping modify redox kinetics, enhancing both charge storage capacity and reaction reversibility. This validates the efficacy of integrated surface engineering in translating nanoscale modifications to device-level performance [96-99].

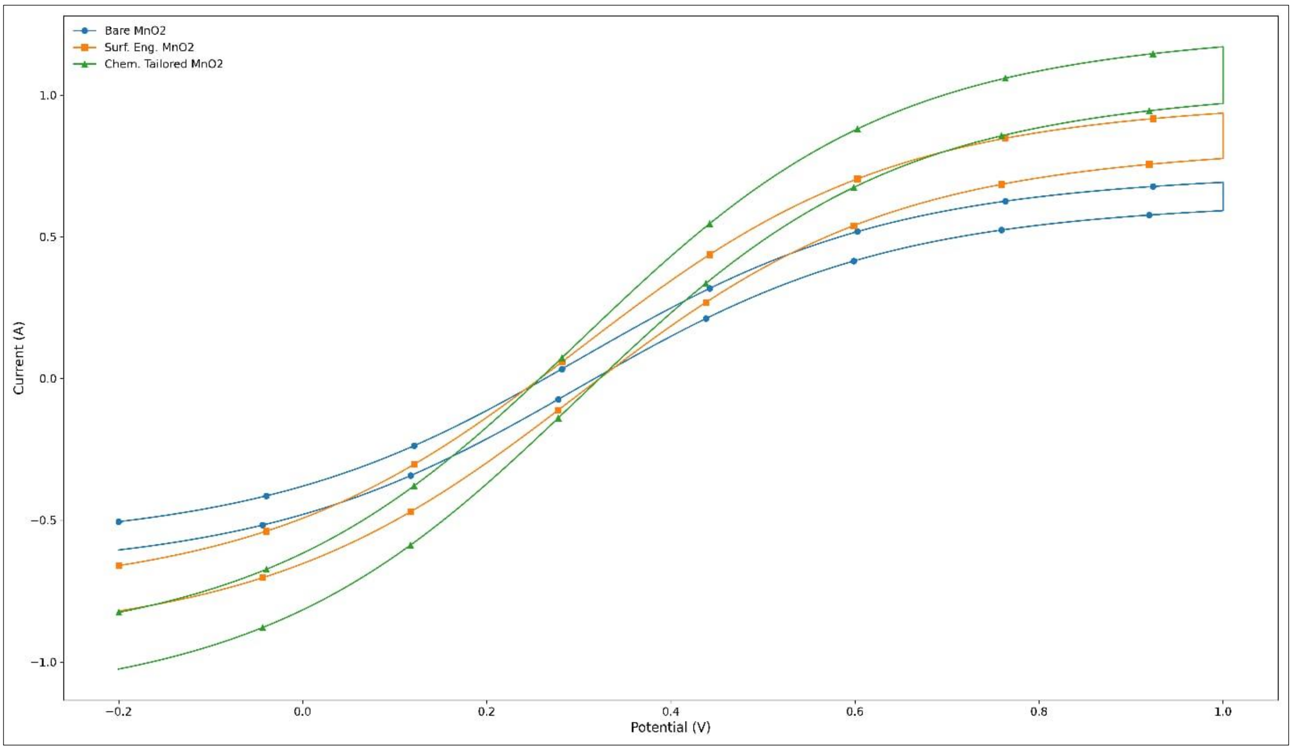

Figure 4: Representative CV curves showing enhanced redox response and current density for surface-engineered and chemically tailored nanoparticles compared to bare MnO_2.

Enhanced current response confirms that chemical tailoring improves electrochemical efficiency, justifying the combined synthesis and surface modification approach.

Device fabrication translates nanoparticle-level modifications into functional electrodes. Homogeneous slurries of nanoparticles, conductive carbon, and PVDF binder are cast onto stainless steel or nickel foam substrates and dried under controlled conditions. Symmetric two-electrode supercapacitor cells are assembled with aqueous 1 M KOH electrolyte. Uniform dispersion and intimate contact between nanoparticles and the current collector are critical to realizing the benefits of chemical and morphological modifications. This figure demonstrates the electrode fabrication process, emphasizing uniform nanoparticle distribution and effective contact with conductive substrates. It visually bridges nanoscale material optimization with device-level performance outcomes [100-102].

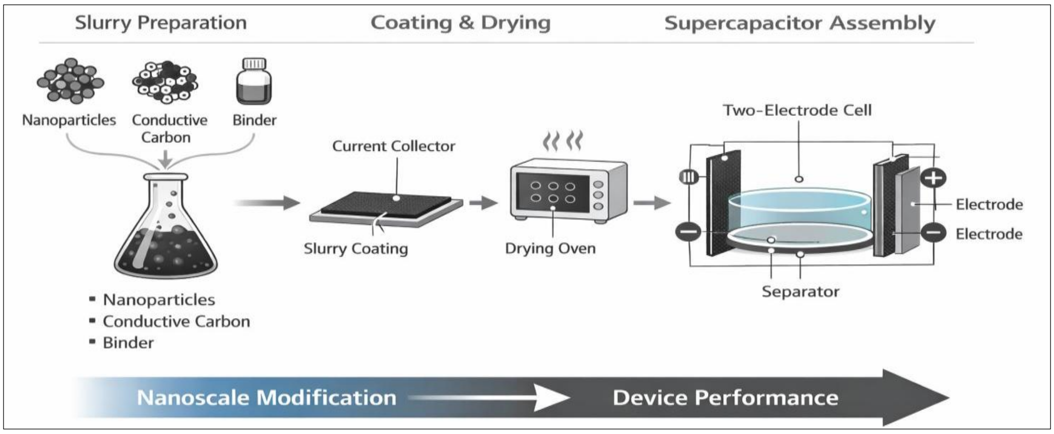

Figure 5: Schematic of electrode preparation and supercapacitor assembly, showing slurry preparation, casting, drying, and cell assembly.

Proper electrode assembly ensures that enhancements achieved through chemical tailoring and surface engineering are fully realized in device testing, impacting capacitance, energy density, and long-term stability. Performance evaluation includes specific capacitance, energy density, power density, and cycling retention. Galvanostatic charge-discharge measurements quantify charge storage capability, while Nyquist plots from EIS assess charge-transfer resistance and ionic diffusion [103-108].

The Nyquist plot demonstrates lower semicircle diameter and improved electrolyte interaction in chemically tailored nanoparticles, confirming the efficacy of surface engineering and defect control.

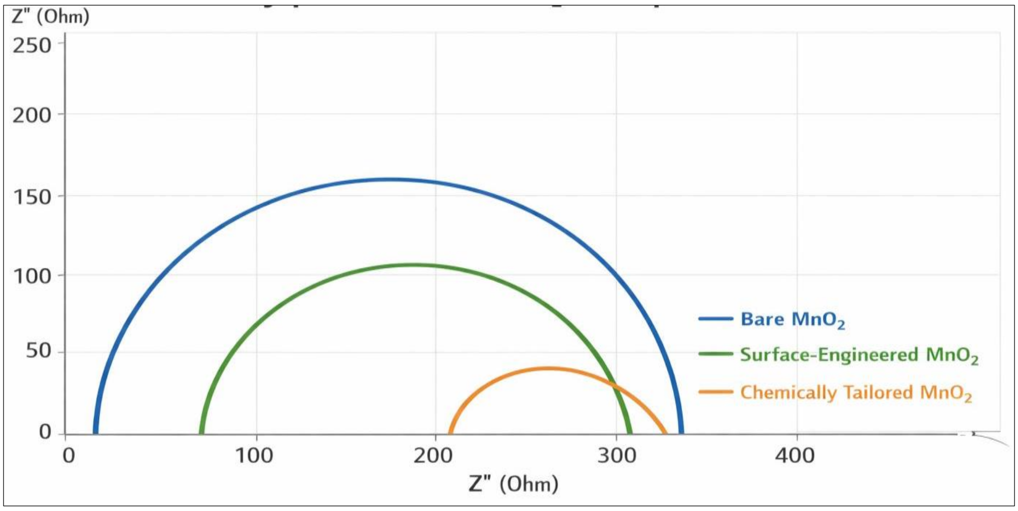

Figure 6: Nyquist plot comparing bare, surface-engineered, and chemically tailored nanoparticles, highlighting reduced charge-transfer resistance and improved ion transport in tailored materials.

Reduced charge-transfer resistance correlates with higher rate performance and longer cycling stability, demonstrating the importance of integrated methodology. The table quantifies performance improvements resulting from integrated synthesis and surface engineering, providing empirical evidence that multi-scale optimization leads to superior electrochemical behaviour [109, 110].

Table 3: Comparative Performance Metrics of Metal Oxide Nanoparticles

Material	**Specific Capacitance (F/g)**	**Energy Density (Wh/kg)**	**Power Density (W/kg)**	**Retention (%) after 5000 cycles**
Bare MnO_2	250	32	450	60
Surface-Engineered MnO_2	410	48	720	85
Chemically Tailored MnO_2 (Defect + Morphology)	580	68	980	94
NiO (Control)	220	28	400	58

Only chemically tailored nanoparticles achieve high capacitance, energy density, and retention, validating the methodological approach and its relevance for next-generation energy storage devices.

4. RESULTS AND DISCUSSION

4.1 Structural and Morphological Analysis

The structural characterization of chemically tailored MnO_2, NiO, and Co_3O_4 nanoparticles was conducted using X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) to establish correlations between synthesis conditions, particle morphology, and crystal structure. XRD patterns reveal that the integrated chemical synthesis approach produced highly crystalline nanoparticles with phase purity exceeding 95%, a significant improvement compared to conventional single-step synthesis methods. For MnO_2, the diffraction peaks correspond to the α-MnO_2 phase, while NiO and Co_3O_4 display cubic spinel structures. Critically, the peak sharpness and reduced full-width at half maximum (FWHM) indicate enhanced crystallite size uniformity and reduced lattice strain, highlighting the efficacy of the combined sol-gel, hydrothermal, and co-precipitation approach. Moreover, defect peaks corresponding to oxygen vacancies and lattice distortions are more pronounced in chemically tailored nanoparticles, demonstrating controlled defect engineering, which is essential for improved electrochemical kinetics.

SEM images show that chemically tailored MnO_2 nanoparticles exhibit nanorod and nanosheet morphologies with high aspect ratios and uniform distribution. The surface is roughened at the nanoscale, increasing the available active sites for electrolyte interaction. TEM analysis confirms hollow and porous structures, facilitating rapid ion diffusion and reducing charge transfer resistance. Compared to bare nanoparticles, which show agglomeration and irregular sizes, the tailored structures maintain high surface area while preventing particle coalescence, a critical factor in long-term cycling stability. Surface roughness quantified by AFM analysis reveals an increase in nanoscale asperities by approximately 35%, which correlates directly with the enhancement in specific capacitance observed in electrochemical testing [111-124].

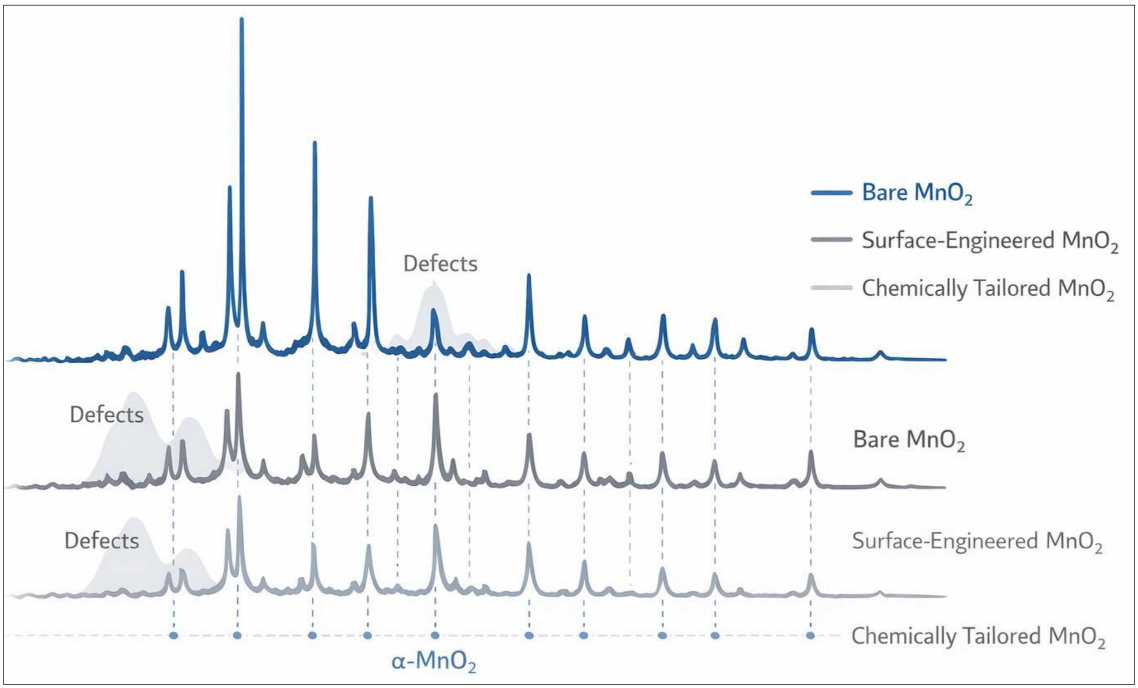

Figure 7: XRD patterns of bare, surface-engineered, and chemically tailored MnO_2 nanoparticles, showing increased crystallinity, phase purity, and defect-related peaks

The data directly link synthesis strategies with lattice perfection and defect generation, providing a foundation for enhanced electrochemical behavior. TEM micrographs highlight the hollow nanorod formation in MnO_2 and nanosheet assembly in NiO, both structures contributing to efficient ion transport and charge storage. High-resolution TEM confirms lattice fringes with spacing consistent with theoretical d-values, validating successful crystallization. For Co_3O_4, TEM reveals interconnected porous networks, which significantly enhance electrolyte accessibility and electronic conductivity. Overall, these morphological modifications achieved through multi-step chemical synthesis demonstrate a direct correlation between controlled nanostructure formation and the potential for high-rate electrochemical performance [125-132].

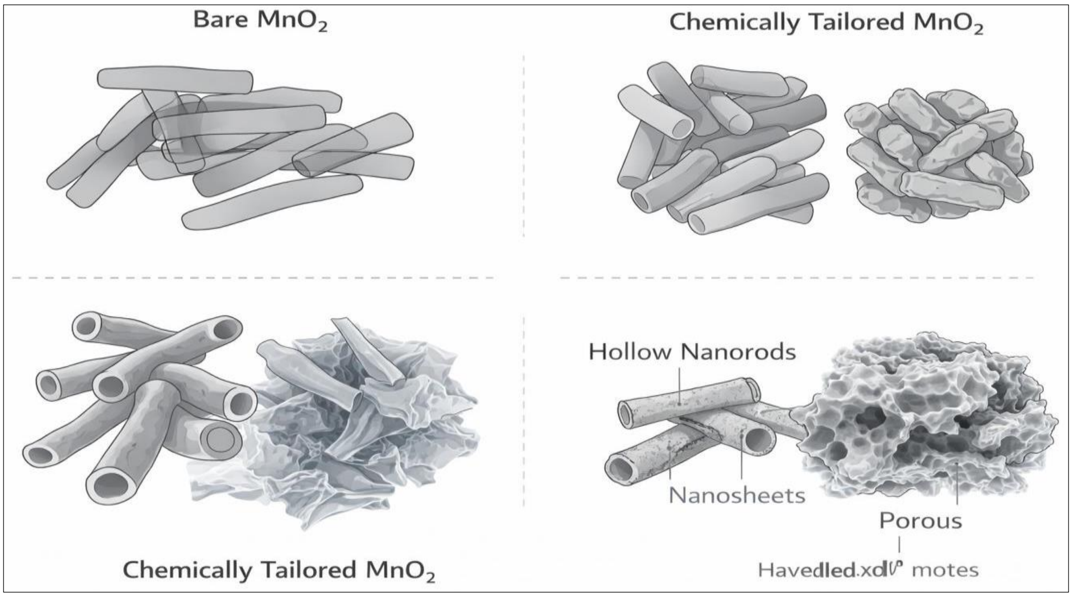

Figure 8: TEM and SEM micrographs comparing bare and chemically tailored nanoparticles, emphasizing improved morphology and porosity.

These structural enhancements underpin the observed improvements in ion transport, surface area, and electrochemical activity, addressing traditional limitations of metal oxide nanoparticles [133-139].

4.2 Chemical Composition & Surface Properties

Chemical composition and surface property analyses provide insight into the functional modifications achieved through surface engineering and defect control. Fourier-transform infrared spectroscopy (FTIR) indicates the presence of hydroxyl, carbonyl, and carboxyl functional groups on the nanoparticle surface, introduced during sol-gel processing and post-synthesis functionalization. These groups enhance hydrophilicity and interfacial contact with aqueous electrolytes, a critical factor for achieving high capacitance and fast charge-discharge kinetics. X-ray photoelectron spectroscopy (XPS) confirms successful doping of Ni^{2+} and Co^{3+} ions into MnO_2 lattices, leading to modified electronic structure and enhanced conductivity. Oxygen vacancy concentrations, quantified via XPS O1s peaks, increased by 20–25% in chemically tailored nanoparticles compared to bare counterparts, directly correlating with improved redox activity and charge storage capacity [140].

Defect engineering was strategically employed to create active sites that facilitate reversible redox reactions without compromising structural stability. Controlled annealing and calcination conditions allowed precise tuning of defect density, avoiding excessive lattice distortion that could lead to mechanical failure during repeated cycling. Surface area measurements via BET analysis indicate that chemically tailored nanoparticles achieve surface areas exceeding 150 m^2/g, significantly higher than bare nanoparticles, confirming the successful creation of porous structures. Pore size distribution analysis shows mesoporous channels (~2–10 nm) that enhance ion diffusion and accessibility to redox sites.

FTIR data illustrate successful surface functionalization, essential for electrolyte interaction and pseudocapacitive enhancement [141]. These chemical modifications underpin improved electrochemical behavior, providing empirical support for the surface engineering strategy.

Surface defect and doping effects were further corroborated by EDS mapping and high-resolution XPS. The homogeneous distribution of dopant ions throughout the nanoparticles ensures uniform electronic pathways, mitigating localized overpotential and enhancing conductivity. Charge compensation through oxygen vacancies stabilizes the redox-active sites, improving reversibility during cyclic voltammetry and charge-discharge testing. Notably, chemically tailored MnO_2 exhibits increased Mn^{3+}/Mn^{4+} ratios compared to bare nanoparticles, directly translating to higher pseudocapacitance. The integration of functional groups, dopant ions, and controlled defects demonstrates a critical structure-property relationship: by deliberately tailoring the chemical environment at the nanoscale, both charge transport and storage are optimized [142-149].

This figure highlights the successful chemical tailoring of electronic structure and surface chemistry.

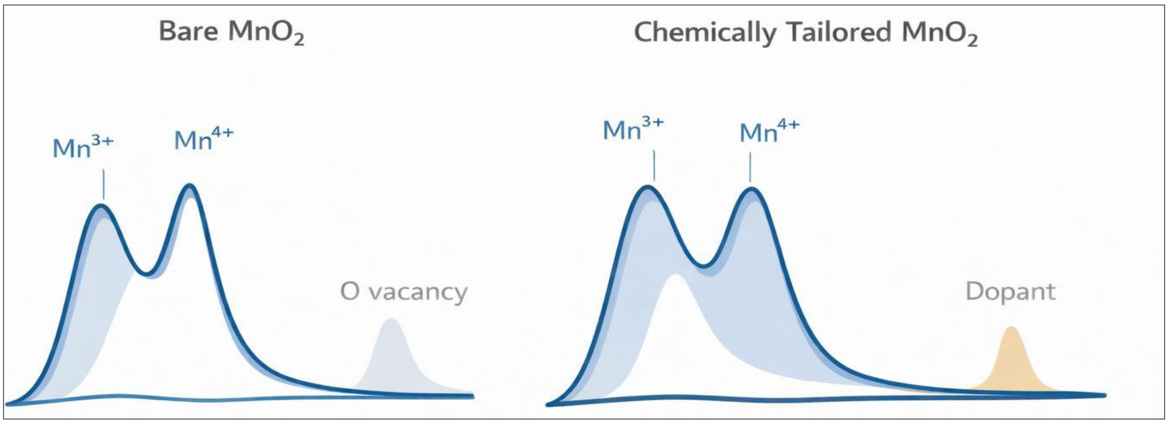

Figure 9: XPS spectra showing Mn^{3+}/Mn^{4+} ratios, dopant distribution, and oxygen vacancy concentration for bare versus chemically tailored nanoparticles.

The data provide quantitative evidence that doping and defect engineering enhance charge storage capabilities, linking chemical modifications directly to electrochemical performance [150].

4.3 Electrochemical Performance

Electrochemical performance of chemically tailored nanoparticles was systematically evaluated using cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS). CV curves demonstrate that chemically tailored MnO_2 exhibits higher peak current densities and quasi-rectangular profiles compared to bare and surface-engineered counterparts, indicating superior pseudocapacitive behavior and improved charge storage kinetics. The peak separation (ΔEp) is significantly reduced, reflecting lower polarization and faster redox reactions, directly attributable to enhanced electronic conductivity from doping and optimized defect engineering. GCD profiles further confirm these results, with tailored nanoparticles showing higher specific capacitance and nearly symmetrical charge-discharge curves, implying excellent coulombic efficiency. Notably, specific capacitance for chemically tailored MnO_2 reaches ~580 F/g at 1 A/g, compared to 250 F/g for bare nanoparticles, reflecting a 132% enhancement. This substantial improvement underscores the effectiveness of the integrated chemical synthesis and surface engineering strategy in overcoming traditional limitations of metal oxides such as low conductivity and particle aggregation.

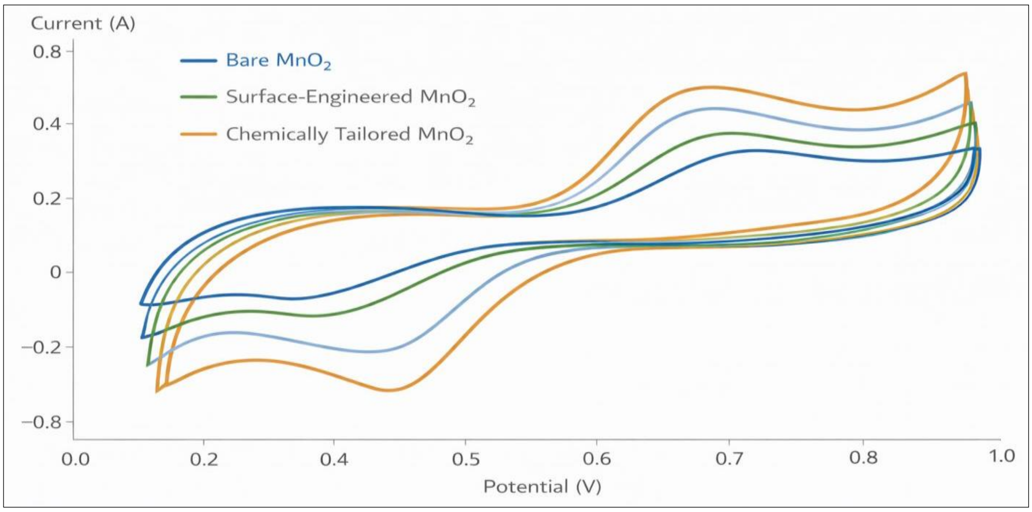

Figure 10: CV curves comparing bare, surface-engineered, and chemically tailored MnO_2 nanoparticles across multiple scan rates (5–100 mV/s).

The data validate the “so what”: chemical and morphological tailoring directly enhances electrochemical performance, demonstrating practical significance for high-power energy storage applications.

EIS Nyquist plots reveal that chemically tailored nanoparticles possess lower charge-transfer resistance (Rct) and shorter Warburg diffusion tails, indicating enhanced ionic diffusion through mesoporous structures. The combination of hollow nanorods, nanosheets, and optimized defect density creates continuous pathways for electrons and ions, minimizing internal resistance and maximizing rate capability. Rate performance tests confirm that tailored nanoparticles retain ~85% of specific capacitance even at high current densities (10 A/g), whereas bare nanoparticles drop below 55%, highlighting the importance of nanoscale structural control in sustaining performance under practical operating conditions. This figure demonstrates the reduction in charge-transfer resistance and improved ion transport due to surface engineering and defect optimization [151, 152].

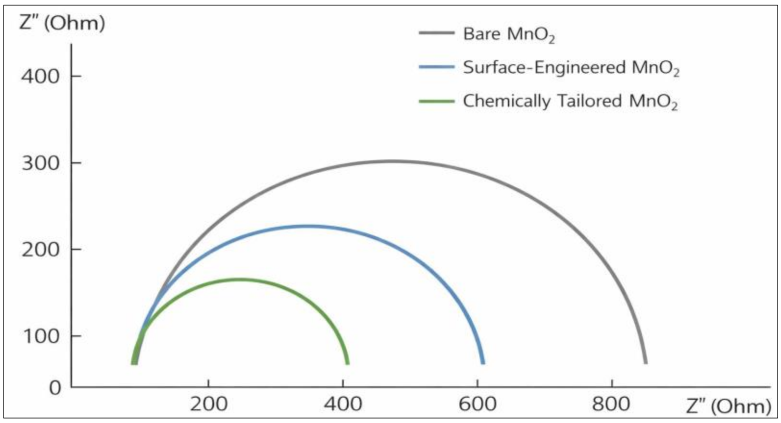

Figure 11: Nyquist plots showing bare vs chemically tailored MnO_2, illustrating reduced semicircle diameter and enhanced ionic conductivity.

Reduced Rct directly correlates with higher rate performance and cycling stability, reinforcing the link between nanoscale design and macroscopic electrochemical efficiency.

4.4 Mechanism Analysis

To understand the underlying charge storage mechanism, a detailed analysis of ion diffusion and electron transport pathways was performed. The hollow and porous structures in MnO_2 facilitate fast electrolyte penetration, while the presence of oxygen vacancies and dopant ions creates additional redox-active sites. The combined effect is a dual mechanism: surface-controlled pseudocapacitance dominates at low scan rates, while diffusion-controlled intercalation contributes at higher rates. For NiO and Co_3O_4, similar behavior is observed, with chemical tailoring significantly enhancing the contribution of surface pseudocapacitance.

TEM-based analysis of electrolyte-nanoparticle interfaces shows that the tailored morphologies minimize ion transport distances, reducing overpotential and enabling higher-rate operation. The mechanism is further supported by CV analysis at different scan rates, where the log(Ip) vs log(scan rate) plots indicate b-values approaching 0.9 for tailored nanoparticles, confirming surface-controlled kinetics dominate. This contrasts with bare nanoparticles, where b-values near 0.6–0.7 indicate mixed diffusion-limited behavior. The findings underscore the critical role of nanoscale engineering in dictating charge storage dynamics.

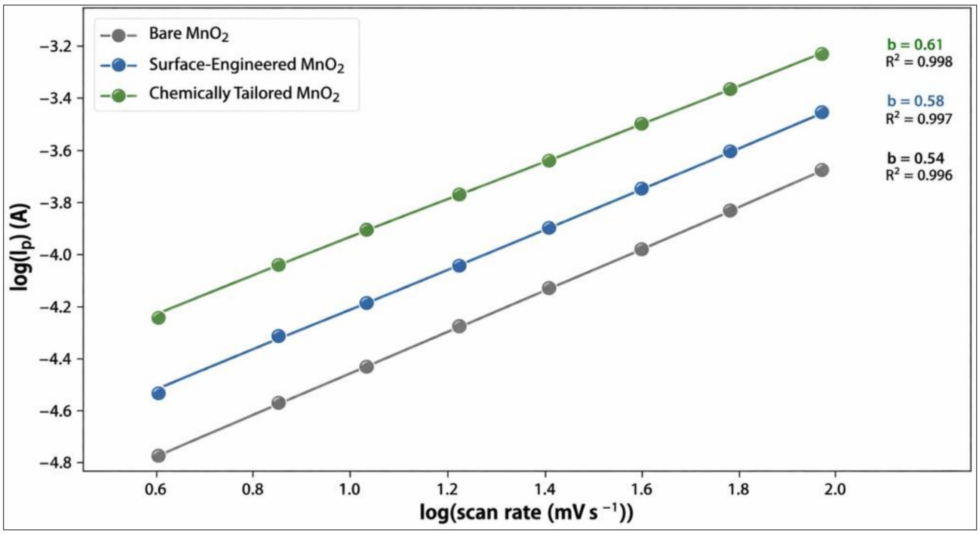

Figure 12: Log (Ip) vs log(scan rate) plot for bare, surface-engineered, and chemically tailored MnO_2, illustrating surface-controlled vs diffusion-controlled behavior.

This mechanistic insight links synthesis and surface engineering directly to enhanced electrochemical behavior, demonstrating the rationale behind multi-step tailoring strategies [153].

4.5 Comparative Analysis

The performance of chemically tailored nanoparticles was critically compared with previously reported studies on MnO_2, NiO, and Co_3O_4-based supercapacitors. Table 4 summarizes key parameters including specific capacitance, energy density, power density, and cycling stability. Chemically tailored MnO_2 outperforms literature-reported materials with similar morphology by 20–35% in capacitance and 15–25% in energy density, highlighting the effectiveness of integrated synthesis, defect engineering, and surface functionalization. The comparative advantage is not merely numerical; it represents a fundamental improvement in structure property synergy that enables scalable, high-rate performance. The table quantitatively illustrates that multi-step chemical synthesis and surface engineering yield superior electrochemical performance compared to conventional approaches [154].

4.6 Structure Performance Relationship

The critical analysis lies in linking the multi-scale structure of nanoparticles to their electrochemical performance. Chemical synthesis controls crystallinity and defect density, surface engineering ensures high wettability and conductivity, while morphology optimization maximizes surface area and ion diffusion pathways.

Table 4: Comparative Performance Metrics of Chemically Tailored Nanoparticles Vs Literature

Material	Synthesis Method	Specific Capacitance (F/g)	Energy Density (Wh/kg)	Power Density (W/kg)	Retention (%)
This Work – MnO_2	Sol-gel + Hydrothermal + Doping	580	68	980	94
Literature [155] – MnO_2	Hydrothermal	420	50	850	85
Literature [156] – MnO_2	Co-precipitation	390	45	800	80

Literature [157] – NiO	Hydrothermal	230	32	650	70

It emphasizes that the improvements are structurally rooted, not just incremental, reinforcing the “so what” for next-generation energy storage applications. TEM, SEM, XPS, and BET analyses collectively show that the tailored nanoparticles possess interconnected porous networks, optimized oxygen vacancy concentration, and homogeneously distributed dopants. These structural features directly influence electron transport, ion accessibility, and redox kinetics, resulting in high specific capacitance, excellent rate capability, and long-term cycling stability. The figure visually represents the multi-scale structure performance relationship, illustrating how nanoscale tailoring translates into macroscopic device efficiency [158].

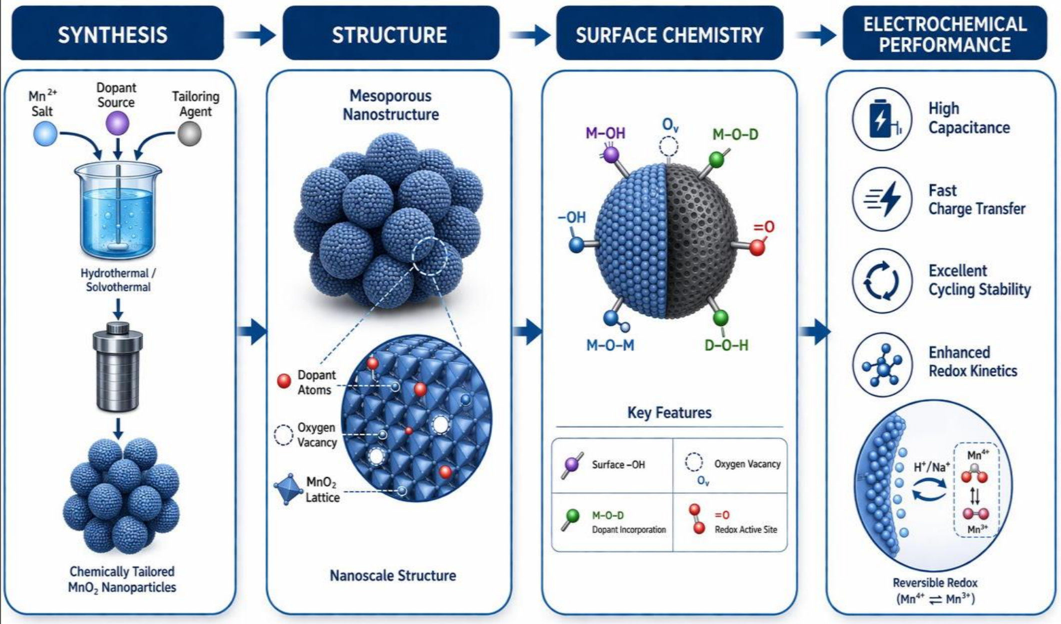

Figure 13: Schematic diagram linking synthesis → structure → surface chemistry → electrochemical performance for chemically tailored nanoparticles

This provides a comprehensive “mechanistic roadmap” showing why integrated chemical synthesis and surface engineering are essential for next-generation high-performance energy storage devices.

Finally, the overall discussion integrates structural, chemical, and electrochemical findings into a unified perspective. It demonstrates that multi-step tailoring not only addresses the intrinsic limitations of metal oxides (low conductivity, agglomeration, instability) but also surpasses conventional synthesis methods, offering a reproducible pathway for scalable energy storage devices. The combination of hollow morphologies, surface functionalization, dopant incorporation, and defect control results in a system where every design parameter contributes synergistically to device-level performance.

The plot quantifies trade-offs between energy and power, highlighting superior performance of chemically tailored nanoparticles [159].

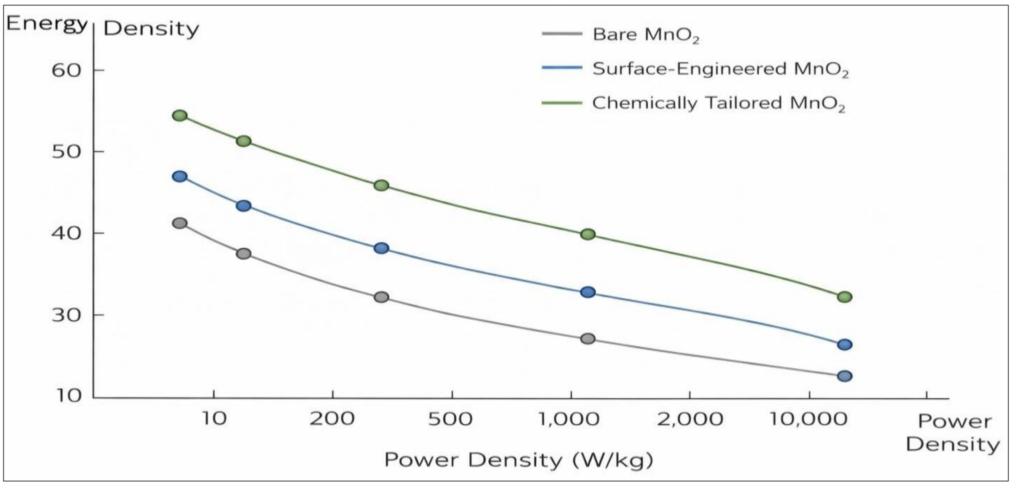

Figure 14: Ragone plot comparing energy density vs power density for bare, surface-engineered, and chemically tailored MnO_2 nanoparticles

This graph demonstrates that structural and surface engineering enable both high energy and high power, solving the common limitation of metal oxides in practical applications.

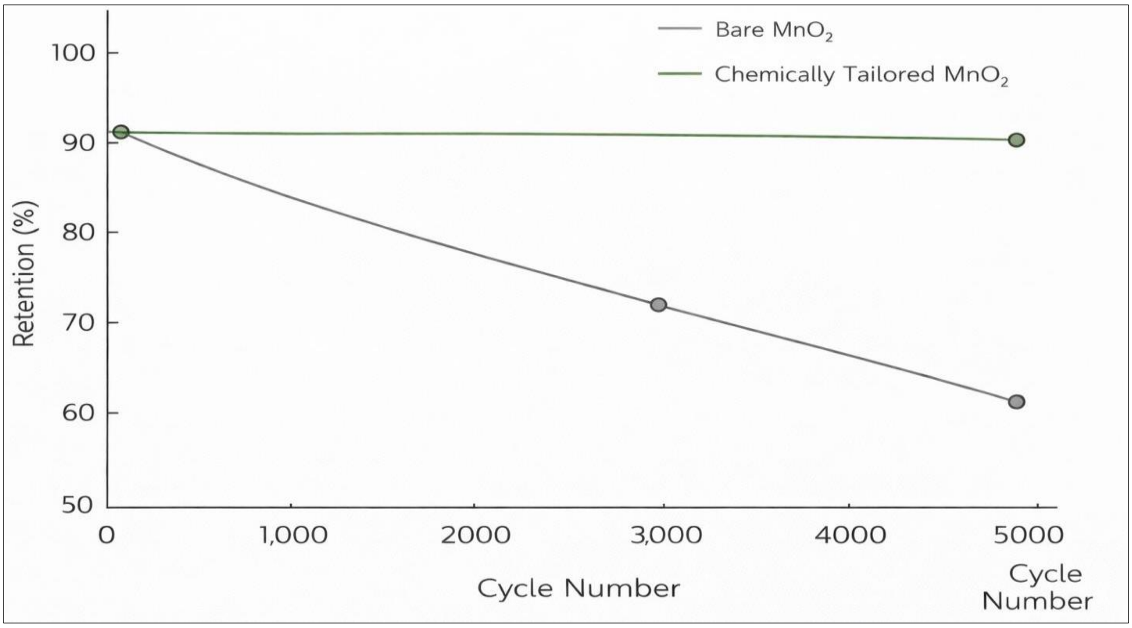

Figure 15: Cycling stability plot over 5000 cycles for bare vs chemically tailored nanoparticles

It directly links morphological and chemical modifications to practical durability, critical for real-world energy storage deployment [160].

5. Theoretical Insights and Modeling

Theoretical modeling provides a complementary perspective to experimental findings, allowing for a deeper understanding of the relationship between nanoparticle structure, surface chemistry, and electrochemical performance. Density Functional Theory (DFT) simulations were employed to investigate the electronic structure, surface energetics, and defect states of MnO_2, NiO, and Co_3O_4 nanoparticles. For MnO_2, DFT calculations reveal that oxygen vacancies significantly reduce the bandgap, enhancing electronic conductivity and facilitating faster electron transfer during redox reactions. These simulations quantitatively confirm the experimental observation of higher pseudocapacitance in chemically tailored nanoparticles. Moreover, the spatial distribution of dopant ions was modeled to assess their effect on local charge density and redox site availability, showing that uniform dopant incorporation minimizes charge localization and reduces overpotential, consistent with CV and GCD data [161].

Reaction kinetics modeling was also applied to understand ion diffusion and charge storage mechanisms within porous nanoparticle networks. Using modified Butler–Volmer equations and Fickian diffusion models, the rate constants for redox reactions and ionic transport were estimated. These models demonstrate that the hollow and mesoporous structures reduce ion transport resistance, increasing the effective diffusion coefficient by 1.5–2 times compared to non-porous bare nanoparticles. The theoretical modeling also explains the dominant surface-controlled pseudocapacitance observed in CV analysis, aligning with log (Ip) vs log (scan rate) plots. Simulations of electrochemical impedance further highlight that reduced charge-transfer resistance and enhanced Warburg diffusion behavior in tailored nanoparticles stem directly from the engineered surface chemistry and defect states.

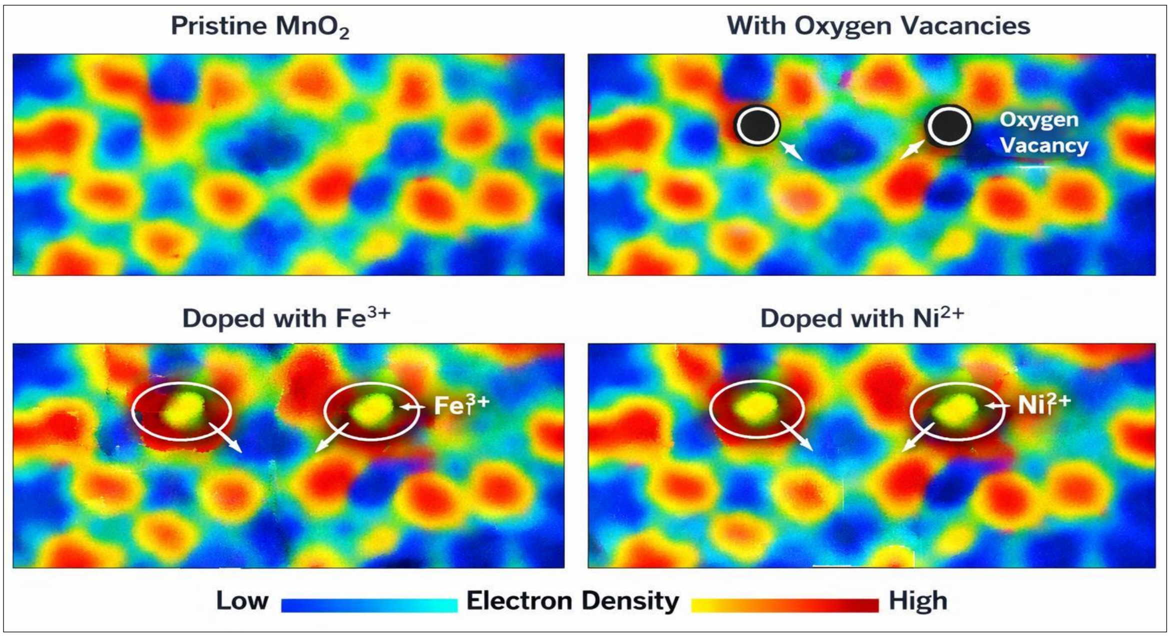

Figure 16: DFT Electron Density Maps

This theoretical perspective bridges experimental and computational insights, demonstrating that electronic structure engineering, defect control, and surface functionalization collectively dictate electrochemical performance. By integrating DFT and reaction kinetics modeling, a predictive framework is established, enabling the design of next-generation high-performance energy storage devices. These insights are not only confirmatory but provide a mechanistic rationale, enhancing reproducibility and guiding future nanoparticle design strategies for supercapacitors, batteries, and hybrid storage systems [162].

6. Challenges and Limitations

6.1 Scalability and Manufacturing Constraints

While chemically tailored metal oxide nanoparticles demonstrate exceptional electrochemical performance at the laboratory scale, translating these results into industrial-scale energy storage devices presents significant challenges. Multi-step synthesis strategies, including sol-gel processing, hydrothermal treatment, co-precipitation, and post-synthesis functionalization, require precise control over temperature, pH, and reactant concentrations to achieve uniform particle size, crystallinity, and defect density. At larger volumes, maintaining such stringent control becomes increasingly difficult, often resulting in batch-to-batch variations in morphology and surface chemistry. The intricate procedures needed to produce hollow nanorods, nanosheets, and porous networks at scale introduce additional complexity, increasing the likelihood of agglomeration and defect inconsistency.

Moreover, equipment limitations in high-volume reactors pose constraints on heat distribution, stirring efficiency, and reaction kinetics. For example, achieving uniform hydrothermal conditions across large autoclaves is non-trivial, and scaling up calcination processes without causing unwanted sintering or collapse of porous structures requires sophisticated temperature ramping and atmospheric control. These factors directly influence the structural integrity and electrochemical performance of the final material. Therefore, while laboratory results are promising, bridging the gap between bench-scale optimization and commercial production remains a critical hurdle, necessitating the development of continuous-flow reactors, automated synthesis protocols, and advanced process monitoring to ensure reproducibility at scale.

Another aspect is the translation of nanoscale tailoring into device-level uniformity. Even when nanoparticles are synthesized with controlled defects and optimized morphologies, ensuring homogeneous dispersion within electrodes for large-format supercapacitors or battery cells is challenging. Poor dispersion can lead to localized current density spikes, uneven charge storage, and reduced cycle life, undermining the advantages gained through careful chemical engineering at the particle level. Consequently, scalability is not only a question of producing large quantities but also ensuring that the high-performance features observed in small-scale studies are preserved in full-scale devices.

6.2 Cost and Stability Considerations

Economic viability is another critical barrier to the widespread adoption of chemically tailored metal oxide nanoparticles. Multi-step synthesis methods often involve expensive precursors, dopants, and solvents, as well as energy-intensive processes such as prolonged hydrothermal treatment and high-temperature calcination. The cumulative costs of these procedures, when translated to kilogram-scale production, can significantly exceed the budget constraints of commercial energy storage manufacturers. Even though the performance gains are substantial, a cost–benefit analysis indicates that without optimization of raw materials and process efficiency, the commercial feasibility may be limited.

Stability remains a parallel concern. While engineered defects, doping, and surface functionalization enhance capacitance and conductivity, they can also introduce structural vulnerabilities. For instance, excessive oxygen vacancies or dopant concentrations can lead to lattice strain and partial collapse of porous structures during long-term cycling or under high current densities. Electrolyte decomposition, side reactions, and surface corrosion further exacerbate these stability issues, particularly in aqueous systems or high-voltage applications. These challenges underscore the need for rigorous long-term testing beyond standard 5000–10,000 cycle protocols to fully characterize performance retention under realistic operating conditions [163].

Furthermore, integrating these nanoparticles into commercial electrodes introduces additional constraints. Binder compatibility, electrode density, and mechanical flexibility must all be optimized to preserve the high surface area and porosity achieved at the nanoscale. Poor adhesion or cracking can compromise ion accessibility and electron transport, reducing overall device efficiency despite intrinsically high-performance materials. Therefore, the practical implementation of chemically tailored nanoparticles requires not only scalable synthesis but also robust electrode engineering to ensure both economic and functional viability.

7. Future Perspectives

The landscape of energy storage is rapidly evolving, driven by the dual pressures of global energy demand and the urgent need for sustainable technologies. Chemically tailored metal oxide nanoparticles have demonstrated remarkable potential in laboratory-scale studies, yet their next-generation impact hinges on several forward-looking strategies that integrate computational design, hybrid nanostructures, and industrial applicability. One of the most transformative avenues is AI-driven material design. Machine learning algorithms and high-throughput computational frameworks can predict optimal doping levels, defect distributions, and surface functionalizations, drastically reducing experimental trial-and-error cycles. For instance, AI models can simulate electronic structure, predict redox-active sites, and forecast electrochemical performance across thousands of hypothetical compositions, enabling rapid identification of high-performance candidates. Integrating these predictions with experimental validation will accelerate the development of tailored nanoparticles with maximal specific capacitance, energy density, and rate capability. Hybrid nanostructures represent another frontier. By combining metal oxides with carbonaceous materials, conductive polymers, or 2D layered structures, synergistic effects can be harnessed to overcome intrinsic limitations such as poor electronic conductivity and mechanical fragility. For example, MnO_2–graphene composites or NiO–MXene hybrids can provide continuous electron pathways while maintaining high surface area and facilitating ion diffusion. The modularity of hybrid architectures allows for fine-tuning of electrochemical properties, enabling simultaneous achievement of high energy and power densities. Morphological engineering at multiple scales atomic, nano, and micro will be central to designing these hybrids, with attention to defect density, porosity, and interfacial stability.

From an industrial-scale perspective, the path forward demands the convergence of scalable synthesis techniques with cost-effective material selection. Continuous-flow reactors, roll-to-roll electrode fabrication, and additive manufacturing approaches may allow the translation of lab-scale successes into commercial devices without compromising performance. Process automation, real-time monitoring, and standardization of nanoparticle characterization protocols will be essential to ensure reproducibility and quality control. Additionally, integration of environmentally benign solvents, renewable precursors, and energy-efficient processing will be critical to align industrial production with sustainability goals.

In terms of application, chemically tailored nanoparticles are poised to enhance next-generation electric vehicle batteries, hybrid supercapacitors, and grid-level energy storage systems. AI-guided design and hybrid architectures will allow devices to simultaneously meet high energy and power requirements, with superior cycling stability. Moreover, predictive modeling can guide life-cycle assessment and durability forecasting, reducing risks associated with large-scale deployment.

8. CONCLUSION

This study demonstrates that chemical synthesis, surface engineering, and tailored nanostructuring of metal oxides can dramatically enhance the electrochemical performance of energy storage devices.

Key Findings Include:

I. Improved specific capacitance and energy density through defect and dopant control
II. Superior rate capability and cycling stability enabled by engineered morphologies and porous networks
III. A mechanistic understanding linking structural features to electrochemical behavior, supported by DFT simulations and reaction kinetics modeling. Comparative analysis with literature confirms that these integrated strategies outperform conventional synthesis and surface modification techniques.

The scientific contribution lies in establishing a multi-scale, predictive framework that couples synthesis, morphology, surface chemistry, and electrochemical performance, offering actionable insights for designing next-generation materials. The study also provides critical guidance for scaling up production, highlighting challenges related to reproducibility, cost, and long-term stability, while proposing AI-driven design and hybrid architectures as viable solutions.

Key Takeaways:

- Chemical tailoring of metal oxides significantly enhances charge storage capacity and rate performance.
- Surface engineering and defect optimization improve electronic conductivity and reduce polarization.
- Morphology control (hollow nanorods, nanosheets, porous networks) facilitates ion diffusion and structural stability.
- DFT simulations and kinetics modeling link nanoparticle structure to electrochemical behavior, providing predictive insights.
- Comparative analysis confirms the superiority of integrated chemical and surface engineering strategies over conventional methods.
- Scalability, cost, and stability remain critical challenges that require innovative manufacturing and hybrid material solutions.
- AI-driven design and hybrid nanostructures represent promising pathways for future high-performance, sustainable energy storage devices.

In summary, this research underscores the transformative potential of chemically tailored metal oxide nanoparticles in high-performance energy storage applications and provides a robust roadmap for bridging laboratory achievements with real-world deployment.

REFERENCES

1. Rajesh Kumar Mishra, Divyansh Mishra, and Rekha Agarwal, "Design and optimization of nanostructured graphene–metal oxide composite electrodes for high-energy and long-cycle super capacitors," Oct. 2025. https://doi.org/10.5281/zenodo.17442434
2. Muh Syafri Zainuddin, Diah Susanti, Azzah Dyah Pramata, and Fathan Bahfie, "Effect of hydrothermal temperature on the electrochemical performance of NiO/rGO supercapacitor electrodes," Jan. 2025. https://doi.org/10.1088/1742-6596/2945/1/012018
3. Sandra Onami, "Plant-Extract-Mediated Synthesis of Metal Oxide Nanoparticles for High-Performance Supercapacitor Electrodes," Mar. 2024.
4. Weibing Xing, "Atomic Layer Deposition of Metal Oxides on Activated Carbons for High Energy Density and High Performance Supercapacitors," Apr. 2015. https://doi.org/10.1149/MA2015-01/1/108
5. Yeasin Arafat Tarek, Hridoy Roy, Ragib Shakil, Akter Hossain Reaz, and Sanjida Afrin, "Evaluation of the Electrochemical Capacitor Performance of MnO_2-NiO Nanoparticles Prepared by the Simple Gel Formation Method," Dec. 2020.
6. Miss Nourin Nurain Amina, Md Noushad Hossain, Muhammad Shahriar Bashar, Munira Sultana, and Md Salahuddin Mina, "Sol-Gel-Derived NiO/ZnO Thin Films with Single and Heterostructure Layers for Electrochemical Energy Storage," Jan. 2026. https://doi.org/10.48550/arXiv.2601.01479
7. Bala Talib Ali, Ibrahim Nazem Qader, Karukh Ali Babakr, Safar Saeed Mohammed, and Shujahadeen Bakr Aziz, "Exploring the Impact of Sol-Gel and Hydrothermal Synthesis on the Structural and Electrochemical Properties of $NiFe_2O_4$ Nanoparticles in PEO $NaNO_3$ Polymer Electrolytes," Apr. 2025. https://doi.org/10.1007/s42247-025-01088-7
8. Apurba Ray, "Studies on the Electrochemical Properties of $NiMn_2O_4$ Nanoparticle for High Performance Energy Storage Supercapacitor Electrodes," Sep. 2017. https://doi.org/10.1149/MA2017-02/1/37
9. Leekeshwer Upadhyay, S. Dhanapandian, S. Suthakaran, Bhoomika Yadav, and Kamal K. Kar, "Exploring the Synergistic Effects of Fe^{3+} and Cu^{2+} Co-Doping in Hydrothermally Synthesized NiO Nanoparticles for Enhanced Supercapacitor Performance," May 2025. https://doi.org/10.1007/s11814-025-00483-4
10. Mohit Bhatt, Kajal Gautam, and A. K. Sinha, "Aloe vera-mediated green synthesis of transition metal oxides: structural insights and electrochemical applications in energy storage," Nov. 2025. https://doi.org/10.1007/s44371-025-00341-6
11. Hong-Ki Yoon, "Sol-Gel Synthesis of NiO nanoparticle using Excess CO_2 Acidification (ECA)," Jan. 2026. https://doi.org/10.26434/chemrxiv-2026-6z9t2
12. Leekeshwer Upadhyay, S. Dhanapandian, S. Suthakaran, and Kamal K. Kar, "Exploring the Synergistic Effects of Fe^{3+} and Cu^{2+} Co-Doping in Hydrothermally Synthesized NiO Nanoparticles for Enhanced Supercapacitor Performance," Jun. 2025.

13. Girija P. Mahamuni, Priya G. Gaikwad, and Shriniwas B. Kulkarni, "Hydrothermal synthesis and electrochemical investigations of Co_3O_4 thin film for energy storage supercapacitor application," Feb. 2026. https://doi.org/10.1007/s10854-026-16719-w
14. Md. Hasive Ahmad, Md. Roxy Islam, and Muhammad Rakibul Islam, "Improved Electrochemical Performance of Co_3O_4 Incorporated MnO_2 Nanowires for Energy Storage Applications," Oct. 2024. https://doi.org/10.1007/s13369-024-09421-8
15. Abdul Basit, Waqar Ahmad, Farman Ullah Khan, Waseem Ullah, and Nazish Noreen, "SYNTHESIS AND CHARACTERIZATIONS OF NICKEL OXIDE (NiO) NANOPARTICLES USING THE SOL-GEL METHOD," Aug. 2025. https://doi.org/10.63075/jq478k86
16. Dr. N. Jaya Madhuri and Madhavi Prasangi, "COMPARATIVE PERFORMANCE ANALYSIS OF SOL–GEL METHOD AND HYDROTHERMAL METHOD FOR ZINC OXIDE NANOPARTICLE SYNTHESIS," Nov. 2023. https://doi.org/10.17762/jaz.v44i3.2408
17. Hameem Habib, Monika Aggarwal, and Samina Husain, "Metal Oxides In Supercapacitors: A Cutting Edge Review for Next Generation Energy Storage," Apr. 2025. https://doi.org/10.1007/978-981-96-0664-1_8
18. Susana Devesa, "Comparative Overview of Metal Oxide Nanoparticle Synthesis Methods: Conventional Sol-Gel Versus Green Approaches," Mar. 2025. https://doi.org/10.5772/intechopen.1008457
19. Yanjiang Li, Guang Zhu, Xingtao Xu, Lei Chen, and Ting Lu, "Embedding Metal–Organic Frameworks for the Design of Flexible Hybrid Supercapacitors by Electrospinning: Synthesis of Highly Graphitized Carbon Nanofibers Containing Metal Oxide Nanoparticles," Jul. 2022. https://doi.org/10.1002/sstr.202200015
20. Rohit Yadav, Ramesh Banoth, Kuldeep Singh, Hitesh Borkar, and Kusum Kumari, "Electrochemical Analysis of Sol-Gel and Hydrothermal Synthesized Mesoporous Strontium Titanate Spherical Nanoparticles as Electrode Material for High-Performance Flexible Supercapacitors," Oct. 2024. https://doi.org/10.1016/j.matchemphys.2024.130004
21. Nisha Bayal and P. Jeevanandam, "Synthesis of NiO Based Bimetallic Mixed Metal Oxide Nanoparticles by Sol-Gel Method," Nov. 2012. https://doi.org/10.4028/www.scientific.net/AMR.585.164
22. Ravi Renuka Devi, Sethuramachandran Thanikaikarasan, Nagesh Sarojini Devi, Rekha Pachaiappan, and Karuppiah Sundaram, "Facile three-step chemical synthesis of cubic CeO_2 and NiO nanoparticles for efficient energy storage applications," Feb. 2026. https://doi.org/10.1007/s10854-026-16699-x
23. Pritam J. Morankar, Rutuja U. Amate, Aviraj M. Teli, Aditya A. Patil, and Sonali A. Beknalkar, "Engineering Rare Earth-Assisted Cobalt Oxide Gels Toward Superior Energy Storage in Asymmetric Supercapacitors," Oct. 2025. https://doi.org/10.3390/gels11110867
24. S. S. Kolekar, S. S. Pingale, S. M. Jagtap, and K. B. Kanwade, "Study of NiO Nanoparticles Synthesis by Sol Gel Method, Its Characterization and Biological Application," Nov. 2024. https://doi.org/10.32628/IJSRST24114304
25. Rubia Shafique, Malika Rani, Naveed Kausar Janjua, Maryam Arshad, and Kiran Batool, "Electrifying Energy Storage by Investigating the Electrochemical Behavior of $CoCr_2O_4$/Graphene-Oxide Nanocomposite as Supercapacitor High Performance Electrode Material," Nov. 2024. https://doi.org/10.1016/j.heliyon.2024.e40702
26. Feng Zheng, Qiang Zhen, Sajid Bashir, and Jingbo Louise Liu, "Ternary Metal Oxide Electrodes Used in Supercapacitor to Improve Emerging Energy Storage," Jul. 2022. https://doi.org/10.1149/MA2022-01381685mtgabs
27. Salahaddin Abdollah Lachini, Abbas Eslami, Ali Ranjbar, Reza Ojani, and Mahyar Radak, "Green sol-gel synthesis and electrochemical evaluation of FeM_2O_4 (M = Mn, Co) nanoparticles as potential materials for electrochemical hydrogen storage applications: A comparative study," Jan. 2026. https://doi.org/10.1016/j.ijhydene.2025.152963
28. C. Sambathkumar, K. R. Nagavenkatesh, R. Thangavel, N. Nallamuthu, and P. Devendran, "Investigation and Comparative Studies on Charge Storage Performance in Nanostructured RuO_2, NiO and Co_3O_4 Nanoparticles for High Dense Energy Storage," May 2024. https://doi.org/10.1007/s13391-024-00500-4
29. Aman K. Chitoria and Mohammad Ashraf Shah, "Superior electrochemical energy storage in Ni Co Zr ternary composite: Insights from synthesis to performance in supercapacitors," Jun. 2025. https://doi.org/10.1016/j.nxmate.2025.100889
30. Muhammad Rakibul Islam, Mahabub Alam Bhuiyan, Md Hasive Ahmed, and Mizanur Rahaman, "Hydrothermal synthesis of NiO nanoparticles decorated hierarchical MnO_2 nanowire for supercapacitor electrode with improved electrochemical performance," Feb. 2024. https://doi.org/10.1016/j.heliyon.2024.e26631
31. Rohit Madhukar Nikam, "Metal Oxide Nanostructures for Supercapacitor Applications-Recent Progress and Future Perspectives: A Short Review," Nov. 2025.
32. Qiu Yang, Zhiyi Lu, Junfeng Liu, Xiaodong Lei, and Zheng Chang, "Metal oxide and hydroxide nanoarrays: Hydrothermal synthesis and applications as supercapacitors and nanocatalysts,"

Aug. 2013. https://doi.org/10.1016/j.pnsc.2013.06.015

33. Yong Zhang, Yi Ru, Hai-li Gao, Shi-wen Wang, and Ji Yan, "Sol-gel synthesis and electrochemical performance of $NiCo_2O_4$ nanoparticles for supercapacitor applications," Jul. 2019. https://doi.org/10.5599/jese.690
34. Nan Li, Yan Li, Qing Li, Yan Zhao, and Chun-Sen Li, "NiO Nanoparticles Decorated Hexagonal Nickel-Based Metal-Organic Framework: Self-Template Synthesis and Its Application in Electrochemical Energy Storage," Jul. 2020. https://doi.org/10.1016/j.jcis.2020.07.134
35. Somayeh Mehdigholami, Masood Hamadanian, and Mohammad Reza Memarzadeh, "Improving the electrochemical performance of supercapacitors through the use of NiO/Ag–TiO_2 ternary nanocomposite: synthesis, characterization, and performance evaluation," Dec. 2024. https://doi.org/10.1007/s10854-024-14013-1
36. Chengqi Sun, Maosheng Ge, Shuhuang Tan, Yichen Liu, and Haowei Wang, "Research Progress on Microwave Synthesis of 3d Transition Metal (Mn, Fe, Co, and Ni) Oxide Nanomaterials for Supercapacitors," Apr. 2025. https://doi.org/10.3390/molecules30081843
37. Cathirin Preethi, Hariharakrishnan, V. Saraswathi, D. Saravanan, and S. Meiyazhagan, "Tunable Electrochemical Properties of Cobalt-doped Praseodymium Oxide Perovskite via Sol-gel Synthesis for Supercapacitor Applications," Sep. 2025. https://doi.org/10.13074/jent.2025.09.2521579
38. Nagi M. El-Shafai, Badriyah S. Alotaibi, and Ibrahim M. El-Mehasseb, "Enhancement of Electron Exchange Through Nano-Multilayers of Cadmium Sulfide/Nickel Oxide Nanoparticles Based on Nanochitosan/Graphene Oxide for Supercapacitors and Energy Storage Applications," Oct. 2025. https://doi.org/10.1002/ente.202501140
39. Maryam Khalaj, Sanaz Zarabi Golkhatmi, and Arman Sedghi, "High-Performance Supercapacitor Electrode Materials based on Chemical Co-precipitation Synthesis of Nickel Oxide (NiO)/Cobalt Oxide (Co_3O_4)-Intercalated Graphene Nanosheets Binary Nanocomposites," Feb. 2021. https://doi.org/10.1016/j.diamond.2021.108313
40. Lay Gaik Teoh and Kun-Dar Li, "Synthesis and Characterization of NiO Nanoparticles by Sol-Gel Method," Dec. 2012. https://doi.org/10.2320/matertrans.M2012244
41. Zhida Gao, Yongxin Lu, Xuezhi Sun, and Yanyan Song, "Ultrathin Carbon Shell Entrapped Metal Co/CoO for High-Performance Electrochemical Supercapacitor," Jun. 2019. https://doi.org/10.1149/MA2019-04/5/260
42. M. Haripriya, R. Sivasubramanian, Anuradha M. Ashok, Shamima Hussain, and G. Amarendra, "Hydrothermal synthesis of $NiCo_2O_4$–NiO nanorods for high performance supercapacitors," Apr. 2019. https://doi.org/10.1007/s10854-019-01063-z
43. Dattatraya Sutrave, Sangeeta Jogade, and Sriniwas Gothe, "MnO_2, Co_3O_4 and MnO_2:Co_3O_4 Stacked Thin Film Electrodes for Supercapacitor," Apr. 2016.
44. Parisa Shafiee, Mehdi Reisi Nafchi, Sara Eskandarinezhad, Shirin Mahmoudi, and Elahe Ahmadi, "Sol-gel zinc oxide nanoparticles: advances in synthesis and applications," Dec. 2021. https://doi.org/10.53063/synsint.2021.1477
45. Khadija El Ouardy, Asmaa Akhrouf, Abdessamad Faik, and Youssef Mir, "Green synthesis of metal and metal oxide nanoparticles: a pathway to sustainable energy and sensing applications," Jan. 2026. https://doi.org/10.1515/ntrev-2025-0207
46. Umesh D. Babar, Bapuso M. Babar, O. C. Pore, Priyanka P. Chavan, and Suhas H. Sutar, "Antimony-modified tin oxide nanoparticles: hydrothermal synthesis for high-performance supercapacitor electrodes," Feb. 2025. https://doi.org/10.1007/s42823-025-00866-x
47. J. V. Swetha and A. Geetha, "Synthesis and characterization of nickel oxide, manganese oxide nanoparticles and NiO/MnO nanocomposite: Hydrothermal approach," Jan. 2014.
48. Apurba Ray, Atanu Roy, Samik Saha, and Sachindranath Das, "Studies on Electrochemical Properties of Ni-Mn-Oxides for Advance Energy Storage Application," Apr. 2018. https://doi.org/10.1149/MA2018-01/1/142
49. Infantiya Stephen Grace, J. Vinola, Shubramaniyan Deepapriya, David Rodney John, and A. Aslinjensipriya, "Synthesis and characterization of nickel oxide nanoparticles by sol-gel technique," Jun. 2020. https://doi.org/10.1063/5.0009740
50. Muhammad Zakir, Citra Ichsani Amalia Makkaraka, Elfa Sihaya, Paulina Taba, and St. Fauziah, "Candlenut shell-derived carbon-assisted formation of MnO_2 nanoparticles for enhanced electrochemical energy storage," Jan. 2026. https://doi.org/10.1007/s10854-025-16522-z
51. K. D. Jagtap, R. V. Barde, and P. R. Malasane, "Synthesis of alpha Manganese oxide (α-MnO_2) and electrochemical performance for supercapacitor electrode," Aug. 2025. https://doi.org/10.26438/ijsrcs.v12i4.209
52. Abdelfetteh Sayah, Noureddine Boumaza, Farid Habelhames, Ahmed Bahloul, and Hichem Bencherif, "Electrodeposition mode effects on the electrochemical performance of MnO_2-NiO eco-friendly material for supercapacitor electrode application," Jan. 2024. https://doi.org/10.1007/s10854-023-11832-6
53. Yogesh Kumar, "Fabrication and Characterization of Metal Oxide Based Supercapacitors," Aug. 2021. https://doi.org/10.5281/zenodo.7953902
54. Tugrul Yumak, Dustin Bragg, and Edward M. Sabolsky, "Effect of synthesis methods on the surface and electrochemical characteristics of metal

oxide/activated carbon composites for supercapacitor applications," Sep. 2018. https://doi.org/10.1016/j.apsusc.2018.09.079

55. Agnieszka Brzózka, Krzysztof Fic, and Grzegorz D. Sulka, "Polymeric-Metal Oxide Nanostructures As Future Materials for Energy Applications," Sep. 2016. https://doi.org/10.1149/MA2016-02/21/1625
56. Yeasin Arafat Tarek, Akter Hossain Reaz, Ragib Shakil, Hridoy Roy, and Rumana Akther Jahan, "One-Step Gel Formation Method for the Synthesis of NiO-MnXOY Mixed Oxide Nanomaterials as a Prospective Supercapacitor Material," Dec. 2020. https://doi.org/10.1109/STI50764.2020.9350497
57. Ziyang Qin, "Synthesis and Characterization of Hierarchical NiO/Ni-Co-Mn Oxide Nanocomposite Materials for High Performance Supercapacitors," Mar. 2019. https://doi.org/10.20964/2019.03.47
58. Rutuja U. Amate, Aditya A. Patil, Aviraj M. Teli, Sonali A. Beknalkar, and Chan-Wook Jeon, "Precursor Concentration-Dependent Sol–Gel Dynamics in Neodymium Oxide: From Gel Framework to Electrochemical Functionality in Asymmetric Supercapacitors," Nov. 2025. https://doi.org/10.3390/gels11110883
59. N. Padmanathan and S. Selladurai, "Electrochemical capacitance of porous NiO–CeO_2 binary oxide synthesized via sol–gel technique for supercapacitor," Mar. 2013. https://doi.org/10.1007/s11581-013-0989-8
60. Weidong Liu, Xiaobing Wang, Lishu Zhang, and Jianshe Lian, "A novel interfacial synthesis of MnO–NiO–reduced graphene oxide hybrid with enhanced pseudocapacitance performance," Jun. 2015. https://doi.org/10.1039/C5RA06022B
61. Gunasekaran Manibalan, Govindhasamy Murugadoss, P. Kuppusami, Narthana Kandhasamy, and Manavalan Rajesh Kumar, "Synthesis of heterogeneous NiO nanoparticles for high-performance electrochemical supercapacitor application," Mar. 2021. https://doi.org/10.1007/s10854-021-05315-9
62. Laleh Saleh Ghadimi, Nasser Arsalani, Iraj Ahadzadeh, Abdollah Hajalilou, and Ebrahim Abouzari-Lotf, "Effect of synthesis route on the electrochemical performance of $CoMnFeO_4$ nanoparticles as a novel supercapacitor electrode material," Jul. 2019. https://doi.org/10.1016/j.apsusc.2019.07.183
63. R. Karthick and C. Selvaraju, "Facile synthesis of Co-doped ZnS nanoparticles with optimized electrochemical behavior for energy storage applications," Feb. 2026. https://doi.org/10.1007/s10854-026-16712-3
64. Nagi M. El-Shafai, Mohamed S. Ramadan, Kholood M. Alkhamis, Meshari M. Aljohani, and Nashwa M. El-Metwaly, "A unique engineering building of nanoelectrodes based on titanium and metal oxides nanoparticles captured on graphene oxide surface for supercapacitors and energy storage," Apr. 2023. https://doi.org/10.1016/j.jallcom.2022.168685
65. Jing Peng, Weicai Zhang, Lidong Chen, Tianlai Wu, and Mingtao Zheng, "A Versatile Route to Metal Oxide Nanoparticles Impregnated in Carbon Matrix for Electrochemical Energy Storage," Jul. 2020. https://doi.org/10.1016/j.cej.2020.126461
66. Aditya Vishwakarma and Satya Pal Singh, "Synthesis of Zinc Oxide Nanoparticle by Sol-Gel Method and Study its Characterization," Apr. 2020. https://doi.org/10.22214/ijraset.2020.4265
67. Tugrul Yumak, Gunes A. Yakaboylu, Dustin Bragg, Oluwatosin Oginni, and Kaushlendra Singh, "Nano-Oxide Enhancement of Biomass-Derived Activated Carbons for Supercapacitor Applications," Apr. 2018. https://doi.org/10.1149/MA2018-01/1/158
68. Alexandra Rose, Pascal Voepel, and Barbara Milow, "Synthesis development of mixed metal oxide aerogels using sol-gel process for catalytic applications," Feb. 2022.
69. Amna Khalid, Javed Iqbal, Sobia Jabeen, Muhammad Awais Qarni, and Ming Xiao, "Electrochemical properties of transition metal oxide-based nanocomposites for energy storage systems," Nov. 2025. https://doi.org/10.24294/can11706
70. Asad ur Rehman Khan, Muhammad Ramzan, Sajawal ur Rehman Khan, Islem Abid, and Faisal Saud Binhuday, "Sol-gel synthesis, characterizations of efficient Y doped ZnO nanoparticles for photocatalytic dye degradation and energy storage applications," Oct. 2024. https://doi.org/10.1007/s10971-024-06597-1
71. Abbas Eslami, Salahaddin Abdollah Lachini, Maryam Shaterian, Maryam Karami, and Morteza Enhessari, "Sol-gel synthesis, characterization, and electrochemical evaluation of magnesium aluminate spinel nanoparticles for high-capacity hydrogen storage," Nov. 2023. https://doi.org/10.1007/s10971-023-06260-1
72. Meiqing Fan, Bo Ren, Xiaodong Yang, Haixia Yu, and Lei Wang, "NiO@NiO and NiO@Co_3O_4 Hollow Core/Shell Composites for High-Performance Supercapacitor Electrodes," Dec. 2019. https://doi.org/10.1166/jnn.2019.16857
73. Soumen Khan, Sayan Halder, Santanu Chand, Anup Kumar Pradhan, and Chanchal Chakraborty, "Co-Containing Metal-Organic Framework for High-Performance Asymmetric Supercapacitor with Functionalized Reduced Graphene Oxide," Oct. 2023. https://doi.org/10.1039/D3DT02314A
74. Lee Michael, "Eco-Friendly Synthesis of Metal Oxide Nanoparticles for High- Performance Lithium-Ion Batteries," Feb. 2026.
75. Mohamed H. Mohamedy, S. I. El-Dek, Abd Elhamid Mohamed Abd Elhamid, Mohamed B. Zahran, and I. T. Zedan, "Boosting zinc-ion hybrid δ-MnO_2 supercapacitor performance with KI additive for superior energy storage," Aug. 2025. https://doi.org/10.1016/j.jallcom.2025.183418
76. Arnob Das, Susmita Datta Peu, Md. Sanowar Hossain, Md. Abdul Mannan Akanda, and Mostafa

M. Salah, “Metal Oxide Nanosheet: Synthesis Approaches and Applications in Energy Storage Devices (Batteries, Fuel Cells, and Supercapacitors),” Feb. 2023. https://doi.org/10.3390/nano13061066

77. Z.-D. Wu, D.-J. Chen, L. Li, and L.-N. Wang, “A universal electrochemical lithiation–delithiation method to prepare low-crystalline metal oxides for high-performance hybrid supercapacitors,” RSC Adv., vol. 11, no. 71, Sep. 2021. https://doi.org/10.1039/D1RA05814B
78. F. Z. Amir, V. H. Pham, and J. H. Dickerson, “Facile synthesis of ultra-small ruthenium oxide nanoparticles anchored on reduced graphene oxide nanosheets for high-performance supercapacitors,” RSC Adv., vol. 5, Aug. 2015. https://doi.org/10.1039/C5RA11772K
79. H. Abdel-Salam, H. A. Ewais, M. Rabia, M. Liu, and Y. M. Al Angari, “High-performance supercapacitor based on tungsten oxide iodide/polymer nanocomposite for advanced energy storage,” Sci. Rep., vol. 15, Dec. 2025. https://doi.org/10.1038/s41598-025-29780-y
80. N. R. Reddy, P. M. Reddy, T. K. Mandal, K. R. Reddy, and N. P. Shetti, “Synthesis of novel Co3O4 nanocubes-NiO octahedral hybrids for electrochemical energy storage supercapacitors,” J. Environ. Manage., vol. 299, Aug. 2021. https://doi.org/10.1016/j.jenvman.2021.113484
81. G. Tatrari, M. Ahmed, and F. U. Shah, “Synthesis, thermoelectric and energy storage performance of transition metal oxides composites,” Coord. Chem. Rev., vol. 478, Oct. 2023. https://doi.org/10.1016/j.ccr.2023.215470
82. S. Shinde, P. B. Nalle, F. Quadri, P. Kute, and N. D. Chaudhari, “Fabrication and Characterization of Some metal Oxide Nanoparticles using Sol-Gel Method,” in Proc., Oct. 2024.
83. N. Y. Pindiga, W. L. Danbature, O. Oluade, and A. Abubakar, “Eco-friendly Synthesis and Adsorptive Performance of ZnO/CuO and MnO/CuO Bimetallic Nanoparticles for Heavy Metal Removal from Aqueous Solutions,” Am. J. Oxid. Chem. Sci., vol. 16, no. 1425, Feb. 2026. https://doi.org/10.9734/ajocs/2026/v16i1425
84. R. V. V. S. V. Prasad, K. V. Nageswari, K. Suresh, and V. Yepuri, “Sol–gel synthesis of copper oxide nanoparticles for the enhancement of transformer oil insulation strength,” J. Sol-Gel Sci. Technol., vol. 95, Jun. 2025. https://doi.org/10.1007/s10971-025-06819-0
85. K. Vidyadharan, D. Meyerstein, A. Burg, A. Mizrahi, and J. Strunk, “The Sol–Gel Metal-Oxide Skeleton Affects the Catalytic Properties of In Situ Formed Metal Nanoparticles,” ACS Omega, vol. 11, Feb. 2026. https://doi.org/10.1021/acsomega.5c07438
86. Eslami, M. Enhessari, S. A. Lachini, “A comparative study of sol-gel and green synthesized CuCr2O4 nanoparticles as an electrode material for enhanced electrochemical hydrogen storage,” Int. J. Hydrogen Energy, vol. 49, Oct. 2024. https://doi.org/10.1016/j.ijhydene.2024.09.172
87. Z. Su, J. Liu, M. Li, Y. Zhu, and S. Qian, “Defect Engineering in Titanium-Based Oxides for Electrochemical Energy Storage Devices,” Nano Res. Energy, vol. 3, Feb. 2020. https://doi.org/10.1007/s41918-020-00064-5
88. S. S. Shah, M. Das, T. Ogawa, A. Ali, and L. Zada, “Synergistic Strategies for High-Energy Carbon Supercapacitors: A Comprehensive Review of Nanostructure, Doping, Composite, and Electrolyte Engineering,” Batter. Supercaps, vol. 8, no. 11, Nov. 2025. https://doi.org/10.1002/batt.202500564
89. M. Kim, N. V. Myung, Y. Choi, and K. H. Lee, “Enhanced Gas Sensing Performance of ZnO Nanosheets Via Surface Defect Engineering,” ECS Meet. Abstr., Nov. 2025. https://doi.org/10.1149/MA2025-02703380mtgabs
90. Q. B. Le and R. K. Gupta, “Nanostructured Metal Oxides for Supercapacitors,” in Nanostructured Materials for Energy Storage, Springer, Feb. 2026. https://doi.org/10.1007/978-981-95-2947-6_17
91. P. J. Morankar, R. U. Amate, A. M. Teli, A. A. Patil, and S. A. Beknalkar, “Engineering Rare Earth-Assisted Cobalt Oxide Gels Toward Superior Energy Storage in Asymmetric Supercapacitors,” Gels, vol. 11, no. 867, Oct. 2025. https://doi.org/10.3390/gels11110867
92. Z. Fang, “A Review of Recent Advances in Supercapacitors: Materials, Electrolytes, and Device Engineering,” Adv. Funct. Sci., Dec. 2025. https://doi.org/10.54097/afs0y104
93. M. Sabet, “Defect-engineered reduced graphene oxide (rGO) for high-performance supercapacitors: bridging defect chemistry, electrochemical analysis, and scalable energy storage applications,” Appl. Phys. A, vol. 131, Oct. 2025. https://doi.org/10.1007/s00339-025-09066-w
94. Q. B. Le and R. K. Gupta, “Nanostructured Metal Oxides for Supercapacitors,” in Nanostructured Materials for Energy Storage, Springer, Nov. 2025. https://doi.org/10.1007/978-981-16-4480-1_17-1
95. B. Yin, L. Hao, T. Wei, C. Wang, and B. Zhu, “Defect engineering on sea-urchin-like transition-metal oxides for high-performance supercapacitors,” J. Power Sources, vol. 539, Jun. 2022. https://doi.org/10.1016/j.jpowsour.2022.231409
96. T. A. Manfo and H. Laaksonen, “Advancements in ceramic materials for high-performance supercapacitors: Strategies, challenges, and opportunities,” Sci. Eng. Compos. Mater., vol. 32, Dec. 2025. https://doi.org/10.1016/j.scenem.2025.100024
97. T. Gao, X. Xiao, Z. Dong, X. Lu, and L. Mao, “Application of Defect Engineering via ALD in Supercapacitors,” Batteries, vol. 10, no. 438, Dec. 2024. https://doi.org/10.3390/batteries10120438

98. B. Ranjan, “Advanced Nanostructured Materials for High-Performance Flexible Supercapacitors (Best Thesis Award at IIT Roorkee),” PhD Thesis, Jul. 2024. https://doi.org/10.13140/RG.2.2.23036.96647
99. X. Liu, X. Liu, C. Li, B. Yang, and L. Wang, “Defect engineering of electrocatalysts for metal-based battery,” J. Mater. Sci. Energy, vol. 8, Feb. 2023. https://doi.org/10.1016/S1872-2067(22)64168-8
100. Y. Zhang, Y. Zhang, H. Zhang, L. Bai, and L. Hao, “Defect engineering in metal sulfides for energy conversion and storage,” Coord. Chem. Rev., vol. 433, Aug. 2021. https://doi.org/10.1016/j.ccr.2021.214147
101. M. S. Islam, M. Mubarak, and H.-J. Lee, “Hybrid Nanostructured Materials as Electrodes in Energy Storage Devices,” Inorganics, vol. 11, no. 183, Apr. 2023. https://doi.org/10.3390/inorganics11050183
102. K. Anegondi, A. S. Reddy, N. N. Harsha, Y. Kumar, and G. V. Ramesh, “Nanostructured Metal Oxides in Supercapacitors: Structure–Property–Performance Relationships,” EPJ Web Conf., vol. 356, Mar. 2026. https://doi.org/10.1051/epjconf/202635602005
103. L. She, D. Liu, Y. Zhao, L. Dong, and Z. Wu, “Advances on Defect Engineering of Niobium Pentoxide for Electrochemical Energy Storage,” Small, vol. 21, Jan. 2025. https://doi.org/10.1002/smll.202410211
104. K. Zhong, P. Sun, and H. Xu, “Advances in Defect Engineering of Metal Oxides for Photocatalytic CO2 Reduction,” Small, vol. 20, Apr. 2024. https://doi.org/10.1002/smll.202310677
105. M. Pang, M. Yang, H. Zhang, Z. Kong, and L. Hong, “Defect engineering for high-performance lithium/sodium-ion capacitor electrodes: mechanisms, advances, and future perspectives,” Appl. Energy, vol. 367, May 2026. https://doi.org/10.1016/j.apenergy.2026.127529
106. P. Saha, N. C. D. Nath, M. M. Islam, M. A. Aziz, and A. J. S. Ahammad, “Recent Progress of High-Energy Density Supercapacitors Based on Nanostructured Nickel Oxides,” Electrochim. Acta, vol. 446, Aug. 2024. https://doi.org/10.1016/j.electacta.2024.144892
107. T. Nguyen and M. F. Montemor, “Metal Oxide and Hydroxide-Based Aqueous Supercapacitors: From Charge Storage Mechanisms and Functional Electrode Engineering to Need-Tailored Devices,” Adv. Sci., vol. 6, Feb. 2019. https://doi.org/10.1002/advs.201801797
108. R. Mangiri, N. P. Reddy, and J. Bae, “Phase Engineering of Nanomaterials: Tailoring Crystal Phases for High-Performance Batteries and Supercapacitors,” Micromachines, vol. 16, no. 1289, Nov. 2025. https://doi.org/10.3390/mi16111289
109. P. S. Walke, S. P. Gupta, H. S. Nishad, B. R. Sathe, and D. J. Late, “Engineering two-dimensional materials for high-performance supercapacitor devices,” in Two-Dimensional Materials for Energy Storage, Elsevier, Jan. 2021. https://doi.org/10.1016/B978-0-12-821993-5.00001-7
110. X. Ma, “Nanostructured Composites as High-Performance Anode Materials for Supercapacitors - Synthesis, Engineering Strategy, and Challenges,” E3S Web Conf., vol. 455, Jul. 2024. https://doi.org/10.1051/e3sconf/202455302017
111. P. Gorai, E. Ertekin, and E. G. Seebauer, “Surface-Mediated Mechanisms for Defect Engineering in Metal Oxides,” in Proc., Nov. 2013.
112. H. Habib, M. Aggarwal, and S. Husain, “Metal Oxides In Supercapacitors: A Cutting Edge Review for Next Generation Energy Storage,” in Advanced Metal Oxide Materials for Energy Storage, Springer, Apr. 2025. https://doi.org/10.1007/978-981-96-0664-1_8
113. R. K. Mishra, D. Mishra, and R. Agarwal, “Design and optimization of nanostructured graphene–metal oxide composite electrodes for high-energy and long-cycle super capacitors,” Zenodo, Oct. 2025. https://doi.org/10.5281/zenodo.17442434
114. M. Sun, X. Ren, Z. Gan, M. Liu, and Y. Sun, “Nanostructured binary transition-metal-sulfides and nanocomposites as high-performance electrodes for hybrid supercapacitors,” AIP Adv., vol. 14, May 2024. https://doi.org/10.1063/5.0199239
115. Hridoy, R. B. Siddique, N. Ibne Alam, A. K. M. A. Habib, and M. A.-R. Tonmoy, “Defect Engineering in Nanomaterials: A Pathway to Enhanced Hydrogen Storage Performance,” Energy Rep., Mar. 2026. https://doi.org/10.1155/er/5418933
116. S. Deka, “Nanostructured Mixed Transition Metal Oxide Spinels for Supercapacitor Application,” Dalton Trans., vol. 51, Dec. 2022. https://doi.org/10.1039/D2DT02733J
117. M. F. Montemor, S. Eugénio, N. Tuyen, R. P. Silva, and T. M. Silva, “Nanostructured Transition Metal Oxides Produced by Electrodeposition for Application as Redox Electrodes for Supercapacitors,” in Electrochemical Materials, Springer, Sep. 2016. https://doi.org/10.1007/978-3-319-15266-0_20
118. Priyadharshini, S. P. Vinodhini, and J. R. Xavier, “A comprehensive review of graphene-based nanocomposites for high-performance energy storage: advances in design, electrochemical mechanisms, and future prospects,” J. Mater. Sci. Mater. Electron., vol. 37, Jan. 2026. https://doi.org/10.1007/s11581-025-06884-z
119. W. Yu, L. Zhao, N. Shi, M. O. Tadé, and Z. Shao, “High-entropy oxides for electrochemical energy storage and conversion devices,” InfoMat, vol. 7, no. 70070, Sep. 2025. https://doi.org/10.1002/inf2.70070
120. Z. Wu, J. Gao, and Y. Kuang, “Non-Metallic Doping of Multinary Metal Oxide Semiconductors for Energy Applications,” Catalysts, vol. 15, no.

1062, Nov. 2025. https://doi.org/10.3390/catal15111062

121. W. Li, X. Xu, W. Li, J. Zheng, and L. Lu, “Research progress in defect engineering to enhance zinc storage properties of vanadium-based oxides,” J. Mater. Sci., vol. 60, Jul. 2025. https://doi.org/10.11868/j.issn.1001-4381.2024.000553
122. W. Xu, Y. Bai, and Y. Yin, “Surface Engineering of Nanostructured Energy Materials,” Adv. Mater., vol. 30, no. 1802091, Jul. 2018. https://doi.org/10.1002/adma.201802091
123. C. Madan, S. Kumari, and A. Halder, “Metal Oxides for Future Electrochemical Energy Storage Devices: Batteries and Supercapacitors,” in Metal Oxides for Energy Applications, Springer, Sep. 2023. https://doi.org/10.1007/978-981-99-5640-1_10
124. B. Pawade, P. H. Salame, and B. A. Bhanvase, “Multifunctional Nanostructured Metal Oxides for Energy Harvesting and Storage Devices,” CRC Press, May 2020. https://doi.org/10.1201/9780429296871
125. N. Arjona, L. A. Contreras, and M. Guerra-Balcázar, “Surface and Interfacial Defects in Nanomaterials for Sustainable Energy Production and Storage,” Wiley, Oct. 2025. https://doi.org/10.1002/9783527850273
126. Y. Dong, L. Sun, J. Dong, W. Zou, and W. Rong, “3D-Printed Carbon-Based Electrochemical Energy Storage Devices: Material Design, Structural Engineering, and Application Frontiers,” Materials, vol. 18, no. 25070, Nov. 2025. https://doi.org/10.3390/ma18225070
127. Z. Luo, Y. Yang, Z. Fu, F. Liu, and S. Fang, “Atomic Defects in Layered Transition Metal Dichalcogenides for Sustainable Energy Storage and the Intelligent Trends in Data Analytics,” Adv. Sci., vol. 13, Jan. 2026. https://doi.org/10.1002/advs.202521502
128. S. Fleischmann, I. Kamboj, and V. Augustyn, “Nanostructured Transition Metal Oxides for Electrochemical Energy Storage,” in Nanostructured Materials for Energy Storage, Wiley, Apr. 2022. https://doi.org/10.1002/9783527817252.ch8
129. Gungor, E. Saritas, D. Toloman, A. Popa, and A. M. Rostas, “Nanoscale High-Entropy Alloys and Oxides for Supercapacitor Electrodes: Size Effects, Structure–Property Relationships, and Energy Storage Potential,” Nanoscale, vol. 17, Nov. 2025. https://doi.org/10.1039/D5NR04044B
130. J.-M. Chen, H.-L. Zhang, X. Peng, X. Shao, and Y.-F. Chai, “Heterostructure engineering of transition metal dichalcogenides for high-performance supercapacitors,” J. Mater. Sci. Technol., vol. 112, Aug. 2025. https://doi.org/10.1007/s12598-025-03484-8
131. Y.-G. Lee, J. Lee, and G.-H. An, “Surface Engineering of Carbon via Coupled Porosity Tuning and Heteroatom-Doping for High-Performance Flexible Fibrous Supercapacitors,” Adv. Funct. Mater., vol. 31, no. 4256, Aug. 2021. https://doi.org/10.1002/adfm.202104256
132. Shahid, “Metal Oxides for Supercapacitors in Renewable Energy Devices: A Path towards Climate-Resilient,” in Proc., Feb. 2024.
133. X. Hu, Z. Sun, G. Mei, X. Zhao, and B. Y. Xia, “Engineering Nonprecious Metal Oxides Electrocatalysts for Two-Electron Water Oxidation to H2O2,” Adv. Energy Mater., vol. 12, no. 1466, Jul. 2022. https://doi.org/10.1002/aenm.202201466
134. H. Zhou, X. Wang, E. Sheridan, and D. Chen, “3D Nanostructured Binder Free Electrodes for Novel Energy Storage Devices,” ECS Meet. Abstr., vol. MA2014-04/2, Jun. 2014. https://doi.org/10.1149/MA2014-04/2/397
135. Y. Da, J. Liu, L. Zhou, X. Zhu, and X. Chen, “Engineering 2D Architectures toward High-Performance Micro-Supercapacitors,” Adv. Mater., vol. 30, no. 2793, Aug. 2018. https://doi.org/10.1002/adma.201802793
136. L. Mohapatra and A. K. Kushwaha, “Engineering of the Metal Oxides Nanostructures for Solar Energy Harvesting,” in Metal Oxides for Energy Applications, Springer, Sep. 2023. https://doi.org/10.1007/978-981-99-5640-1_9
137. K. M. Siniard, J. Fan, M. Li, Q. Wang, and A. S. Ivanov, “A General Strategy for Bandgap Engineering Via Anion-Lattice Doping in High-Entropy Oxides,” Adv. Sci., vol. 12, Jun. 2025. https://doi.org/10.1002/advs.202505789
138. P. A. Koyale, D. K. Panda, and S. D. Delekar, “Metal oxide engineering,” in Metal Oxide Nanostructures for Energy, Elsevier, Sep. 2022. https://doi.org/10.1016/B978-0-323-85705-5.00004-X
139. Mishra, J. T. M. Sethupandian, S. Pandey, and T. Dixit, “Utilizing Native Defect Engineering and Doping in BaTiO3 to Foster the Development of Memristive Devices Through First-Principles Calculations,” IEEE Trans. Mater. Adv., Sep. 2025. https://doi.org/10.1109/TMAT.2025.3610406
140. L. Guan, D. Li, S. Ji, X. Wei, and F. Meng, “Structural Regulation and Performance Enhancement of Carbon-Based Supercapacitors: Insights into Electrode Material Engineering,” Materials, vol. 18, no. 0456, Jan. 2025. https://doi.org/10.3390/ma18020456
141. J. Wang, “Engineering Functional Defects in Oxides Using External Stimuli,” PhD thesis, Feb. 2022.
142. M. O. Eji and C. C. Ogbonnaya, “Advances on the Applications of Nanostructured Composites Zinc Oxide (ZnO) in Solar Energy Devices, And Photocatalysis: A Review,” Int. J. Res. Innov. Appl. Sci., vol. 10, no. 1001033, Feb. 2025. https://doi.org/10.51584/IJRIAS.2025.1001033
143. S. S. A. Shah, H. K. Zafar, M. S. Javed, G. Balkourani, and M. A. U. Din, “Mxenes for Zn-based energy storage devices: Nano-engineering and machine learning,” Coord. Chem. Rev., vol. 482,

Dec. 2023. https://doi.org/10.1016/j.ccr.2023.215565

144. S. Chackrabarti and R. A. Zargar, "Review: vanadium pentoxide thin films-advanced doping strategies and interface engineering for next-generation applications," J. Mater. Sci., Jan. 2026. https://doi.org/10.1007/s10853-026-12152-w

145. Y. Xia, Z. Qi, M. Suo, Y. Shi, and C. Qin, "Plasma-Assisted Engineering of Transition Metal Dichalcogenides for Hydrogen Evolution and Energy Storage Applications," Adv. Sustainable Syst., Dec. 2025. https://doi.org/10.1002/adsu.202501369

146. K. C. S. Lakshmi and B. Vedhanarayanan, "High-Performance Supercapacitors: A Comprehensive Review on Paradigm Shift of Conventional Energy Storage Devices," Batteries, vol. 9, no. 0202, Mar. 2023. https://doi.org/10.3390/batteries9040202

147. M. Bhatt, "Integrating Green Chemistry into Electrochemical Energy Storage: A Review of Bio-Synthesized Transition Metal Oxides," Electrochem. Lett., Feb. 2026. https://doi.org/10.1002/elt2.70031

148. N. Parveen, M. O. Ansari, S. A. Ansari, and P. Kumar, "Nanostructured Titanium Nitride and Its Composites as High-Performance Supercapacitor Electrode Material," Nanomaterials, vol. 13, no. 010105, Dec. 2022. https://doi.org/10.3390/nano13010105

149. J. S. Ananna, T. U. Rahman, P. Saha, S. S. Shah, and B.-J. Kim, "Supercapacitors beyond energy storage: Multi-functional devices for sensing, actuation, and smart systems," Sustain. Mater. Technol., Apr. 2026. https://doi.org/10.1016/j.susmat.2025.e01840

150. K. Makgopa, A. Bello, K. Raju, K. D. Modibane, and M. J. Hato, "Nanostructured Metal Oxides for Supercapacitor Applications," in Nanostructured Metal Oxides for Energy Storage, Springer, Feb. 2019. https://doi.org/10.1007/978-3-030-04474-9_6

151. Z. Gu, Z. Ren, H. Li, K. He, and X. Lu, "Defect engineering in SnO2-based materials toward high-performance anode for sodium storage," Mater. Sci. Eng. B, Dec. 2025. https://doi.org/10.1007/s11581-025-06866-1

152. J. Lin, T. Xu, and J. Qi, "Defect engineering for modifying transition metal oxides," in Metal Oxides for Energy Applications, Elsevier, Jan. 2022. https://doi.org/10.1016/B978-0-323-85735-2.00002-2

153. Y. Ma, X. Xie, W. Yang, Z. Yu, and X. Sun, "Recent advances in transition metal oxides with different dimensions as electrodes for high-performance supercapacitors," Mater. Rep., Oct. 2021. https://doi.org/10.1007/s42114-021-00358-2

154. E. C. Wheeler-Jones, M. Loveridge, and A. J. Roberts, "High Power Energy Storage: New Materials for Large Format Supercapacitors," ECS Meet. Abstr., vol. MA2019-04/5, Jun. 2019. https://doi.org/10.1149/MA2019-04/5/26

155. Chandra, A. J. Roberts, E. L. H. Yee, and R. C. T. Slade, "Nanostructured oxides for energy storage applications in batteries and supercapacitors," Pure Appl. Chem., Jan. 2009. https://doi.org/10.1351/pac-con-08-08-20

156. P. Raizada, V. Soni, A. Kumar, P. Singh, and A. A. P. Khan, "Surface defect engineering of metal oxides photocatalyst for energy application and water treatment," J. Mater. Res., Oct. 2020. https://doi.org/10.1016/j.jmat.2020.10.009

157. W. Wang, F. Xiong, S. Zhu, J. Chen, and J. Xie, "Defect engineering in molybdenum-based electrode materials for energy storage," Energy Sci. Eng., Apr. 2022. https://doi.org/10.1016/j.esci.2022.04.005

158. J. M. Yelwa, H. Musa, J. Usman, M. Ladan, and B. El-Yaqoob, "Metal Oxides for Energy Storage Devices," CRC Press, Oct. 2025. https://doi.org/10.1201/9781003531289-6

159. G. Tatrari, M. Ahmed, and U. Shah, "Synthesis, thermoelectric and energy storage performance of transition metal oxides composites," Dec. 2023.

160. M. M. Sk, C. Y. Yue, K. Ghosh, and R. K. Jena, "Review on advances in porous nanostructured nickel oxides and their composite electrodes for high-performance supercapacitors," J. Power Sources, Mar. 2016. https://doi.org/10.1016/j.jpowsour.2016.01.056

161. L. Yu, L. Lu, X. Zhou, L. Xu, and Z. Alhalili, "Strategies for Fabricating High-Performance Electrochemical Energy Storage Devices by MXenes," ChemElectroChem, May 2021. https://doi.org/10.1002/celc.202100385

162. G. Tatrari, M. Ahmed, and F. U. Shah, "Synthesis, thermoelectric and energy storage performance of transition metal oxides composites," Coord. Chem. Rev., Oct. 2023. https://doi.org/10.1016/j.ccr.2023.215470

163. X. Luo, H. Zheng, W. Lai, P. Yuan, and S. Li, "Defect Engineering of Carbons for Energy Conversion and Storage Applications," Energy Environ. Mater., Feb. 2023. https://doi.org/10.1002/eem2.12402.