

GREEN SYNTHESIS OF TRANSITION METAL OXIDE NANOPARTICLES FOR HIGH-PERFORMANCE ELECTROCHEMICAL ENERGY STORAGE APPLICATIONS

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Abstract

The escalating demand for efficient, eco-friendly energy storage systems has driven the adoption of green synthesis methods for transition metal oxide (TMO) nanoparticles, replacing conventional toxic chemical routes with biogenic approaches using plant extracts, microbes, and algae. This review explores the synthesis, characterization, and electrochemical performance of TMOs such as MnO₂, NiO, Co₃O₄, Fe₂O₃, and ZnO, highlighting their applications in supercapacitors (specific capacitances up to 2000 F/g), lithium-ion batteries (capacities >1000 mAh/g), and sodium-ion batteries. Green routes leverage natural reducing agents (polyphenols, alkaloids) for controlled nanoparticle morphology (spheres, rods, sheets), yielding high surface area (50–300 m²/g), enhanced conductivity via doping, and superior cycle stability (>90% retention after 5000 cycles). Mechanisms involve microbial extracellular enzymes and plant-mediated reduction, offering scalability and reduced environmental impact. Challenges include batch variability and purity, addressed through optimized protocols and hybrid composites (e.g., TMO-graphene). The integration of these nanomaterials promises sustainable, high-performance devices for electric vehicles, renewables, and portable electronics, aligning with global green chemistry goals.

1. INTRODUCTION

The global transition toward sustainable energy platforms has necessitated the development of advanced energy storage systems that are not only efficient and high-performing but also environmentally benign (Adeyinka et al., 2024). As population growth drives urbanization and industrial expansion, the demand for energy storage in sectors such as electric vehicles, portable electronics, and grid-scale storage

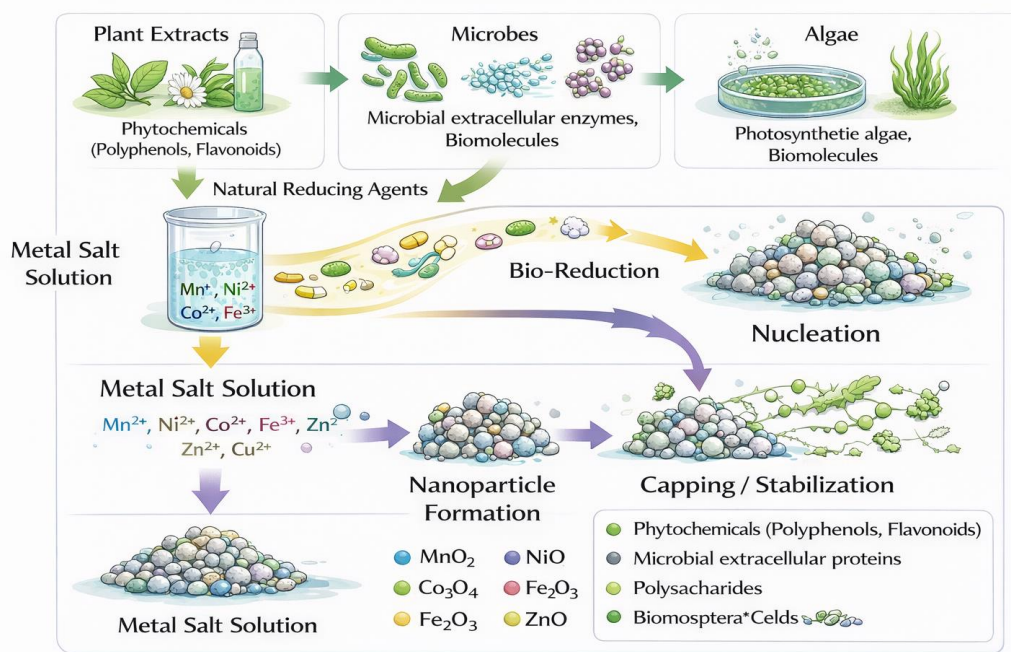
continues to rise, leading to a concurrent increase in chemical waste and environmental degradation (Ekechukwu et al., 2024). Traditionally, the synthesis of transition metal oxide (TMO) nanoparticles, which serve as the cornerstone of modern electrochemical capacitors and batteries, has relied on physical and chemical methodologies that utilize hazardous reducing agents, organic solvents, and high-energy processes (Parveen et al., 2025). The

shift toward biogenic synthesis represents a paradigm change, aiming to replace carcinogenic and toxic substances with renewable biological resources (Nwachukwu et al., 2023).

Transition metal oxides are uniquely suited for energy storage applications due to the proximity in the energy of their d and ns shells, which allows these elements to maintain multiple oxidation states. This electronic flexibility facilitates various configurations for their oxides, providing high theoretical capacities and tunable electrochemical properties (Malavekar et al., 2024). However, the performance of these materials is inextricably linked to their structural and morphological characteristics at the nanoscale. Nanoscale TMOs exhibit high surface-to-volume ratios and increased reactivity compared to their bulk counterparts, enabling faster ion diffusion and superior charge transfer kinetics (Kiruthika et al., 2022). The adoption of

green synthesis addresses the environmental and economic limitations of traditional routes. By utilizing plant extracts, microorganisms, and agricultural waste as reducing and capping agents, this approach minimizes waste, reduces pollution, and operates under ambient conditions (Saratale et al., 2018). Furthermore, the integration of biological molecules often imparts unique surface properties and defects in the crystalline structure of the nanoparticles, which can enhance their electrochemical reactivity and stability beyond what is typically achieved through conventional chemical reduction (Osman et al., 2024). Figure 1 illustrates the green synthesis pathways of transition metal oxide nanoparticles, highlighting plant-mediated, microbial, and algal routes that replace toxic chemicals with eco-friendly reducing agents. The schematic emphasizes the stepwise conversion from metal salts to stabilized nanoparticles using natural biomolecules.

Figure 1: Green Synthesis Pathways of Transition Metal Oxide Nanoparticles



2. Fundamental Principles of Sustainable Nanomaterial Fabrication

The fabrication of transition metal oxide nanoparticles generally follows two primary strategies: the top-down and bottom-up

approaches. Understanding the distinction between these pathways is critical for evaluating the efficiency and sustainability of the resulting energy storage materials. In the top-down approach, larger bulk materials are physically or

chemically disintegrated into nanoscale particles (Palagati et al., 2024). Common techniques include mechanical milling, which uses high-energy ball milling to break down structures. While effective for creating homogeneous mixtures and nanocomposites, it often results in polydisperse particles with poor control over morphology and can be energy-intensive (Tripathy et al., 2023). Lithography, utilizing focused light or particle beams, allows for the creation of intricate patterns on surfaces. While precise, it is often too expensive and slow for the mass production required in the energy sector (George et al., 2025). Laser ablation vaporizes a target material with high-energy laser pulses. Although it eliminates the need for stabilizing chemicals, the high cost of equipment and

maintenance limits its accessibility for large-scale industrial use (Ravi-Kumar et al., 2019).

The bottom-up approach involves the controlled assembly of molecules or atoms to form nanoparticles, offering superior control over size, shape, and chemical composition. Conventional chemical methods such as sol-gel, co-precipitation, and hydrothermal synthesis belong to this category but frequently involve toxic precursors like sodium borohydride or hydrazine (Hachem et al., 2022). Green synthesis, a subset of the bottom-up strategy, utilizes biological reductants derived from plants, fungi, bacteria, and algae. Compared to traditional synthesis, green synthesis allows a 30% reduction in energy consumption, cost savings of up to 40%, and a 50% increase in production output (Badruhisham et al., 2024).

Table 1: Comparison of Nanoparticle Synthesis Methodologies

Property	Green Synthesis	Chemical/Physical Synthesis
Reducing Agents	Natural compounds (e.g., flavonoids, polyphenols)	Toxic chemicals (e.g., NaBH ₄ , hydrazine)
Stabilizers/Capping Agents	Plant metabolites, proteins, polysaccharides	Surfactants, polymers, PEG, CTAB
Reaction Conditions	Mild (ambient temperature and pressure)	Often harsh (high temp, pressure, pH)
Environmental Impact	Minimal, eco-friendly, biodegradable	High, generates hazardous byproducts
Yield	Moderate to low	High
Cost	Low, uses renewable and abundant resources	High due to synthetic reagents and energy
Safety	Benign and safe	Potential hazards or carcinogenicity

3. Biogenic Synthesis Mechanisms and the Role of Phytochemicals

The mechanism by which biological extracts transform metal salts into oxide nanoparticles is an intricate biochemical process involving reduction, nucleation, and stabilization. In plant-mediated synthesis, the primary agents are secondary metabolites, including polyphenols, flavonoids, terpenoids, and organic acids (El Shafey, 2020). Phytochemicals contain functional groups such as hydroxyl (-OH) and carbonyl (C=O) that act as electron donors (Adeyemi et al., 2022). During the one-step synthesis, these

biomolecules facilitate the redox reaction by donating electrons to the metal cations in the precursor solution. For example, in the synthesis of iron oxide nanoparticles, phenolic hydroxide groups form complexes with Fe²⁺ or Fe³⁺ ions, reducing them to their nanoparticle form (Mohamed et al., 2023).

Phytochemical-mediated reduction occurs via electron donation from hydroxyl or carbonyl groups to metal ions. Stabilization arises from the adsorption of phytochemicals onto nanoparticle surfaces, forming a protective layer that prevents aggregation (Mishra et al., 2025). Functional

groups such as -OH, -COOH, and -C=O are critical in these processes. A unique feature of green synthesis is the dual role played by phytochemicals as both reducing and capping agents (Kaouthar, 2022). Once the nanoparticles are formed, biological molecules such as proteins, sugars, and flavonoids adsorb onto the surface of the nanostructures. This capping mechanism is critical because it prevents the agglomeration of the resulting nanoparticles, thereby ensuring they remain uniformly sized and well-defined (Perumalsamy et al., 2024). Biologically capped nanoparticles often exhibit higher reactivity and biocompatibility compared to their chemically synthesized counterparts due to the presence of organic molecules and inherent defects in their crystalline structure (Sysak et al., 2023).

The synthesis of CuO NPs using plant extracts involves several biochemical interactions where biomolecules facilitate the conversion of the metal precursor into the desired nanoparticles (Sidhu et al., 2022). Extreme temperatures can cause the degradation of these bioactive compounds, which limits their ability to reduce and stabilize the particles, leading to larger sizes. The pH of the solution is a critical parameter, as it influences the shape, size, and synthesis rate of the developed nanoparticles (Gebre, 2023). Nucleation center formation is highly dependent on pH; an increase in pH leads to an enhancement in the nucleation center, increasing the metal ion reduction to nanoparticles. Smaller-size nanoparticles typically occur more in a basic environment than in an acidic solution (Shi et al., 2020). For example, in the synthesis of Au NPs using *Aspergillus terreus*, the shape of nanoparticles transforms from spherical to rod at pH 8, whereas at pH 10, the mean size decreases and shapes become spherical (Radulescu et al., 2023).

4. Synthesis and Performance of Manganese Dioxide Nanomaterials

Manganese dioxide (MnO₂) nanomaterials stand out among other materials owing to their good environmental compatibility, low cost, and strong oxidative and adsorptive properties. MnO₂ has different spatial structures and crystalline forms, mainly alpha-MnO₂, beta-MnO₂, gamma-MnO₂,

delta-MnO₂, and lambda-MnO₂ (Yang et al., 2021). The surface physicochemical characteristics vary considerably based on the crystal structure, which can be categorized into one-dimensional (1D) tunnel structures, two-dimensional (2D) layered structures, or three-dimensional (3D) network structures (Narayan, 2023). The synthesis method crucially impacts electrochemical performance; chemical methods may yield materials with poor performance due to side reactions, whereas hydrothermal methods yield crystal structures favorable for charge transfer and ion diffusion (Dawadi et al., 2020).

Green tea-mediated MnO₂ (GT-MnO₂) had a capacity of 198 mAh/g, while black tea (BT-MnO₂) reached 236 mAh/g. MnO₂ synthesized via orange peel (OP) showed an initial capacity of 119 F/g at the first cycle, peaked at 137 F/g (115%) by the 350th cycle, and returned to 119 F/g after 500 cycles, demonstrating an interesting capacity rise phenomenon (Gul et al., 2025). Biogenic synthesis of MnO₂ nanoparticles and MnO₂/eggshell nanocomposites was successfully achieved using aqueous leaf extract of *Ocimum basilicum*. The size of green-synthesized NPs calculated by the Debye-Scherrer equation was 59.46 nm, while the nanocomposites were 32 nm (Ding et al., 2029).

Amorphous manganese dioxide (a-MnO₂) as a cathode in sodium-ion batteries (SIBs) possesses a high theoretical capacity of 308 mAh/g, low cost, and high-rate capability due to its small particle size (5 to 50 nm). The first specific discharge capacity of the amorphous MnO₂ electrode is 123.2 mAh/g and remains at 136.8 mAh/g after 100 cycles at a current rate of 0.1 C (Farhan et al., 2025). The layered delta-MnO₂ with larger interlayer spacing facilitates Zn²⁺ storage and transport in aqueous zinc-ion batteries (AZIBs), but the structure can collapse with repeated insertion/extraction, leading to decay in cycling stability (Zhang et al., 2024). Pre-intercalation of Na⁺ can broaden the layer-to-layer distance and serve as a column support, improving the ion transport rate and stability (Khamsanga, 2020).

5. Nickel and Copper Oxide Nanoparticles for Energy Applications

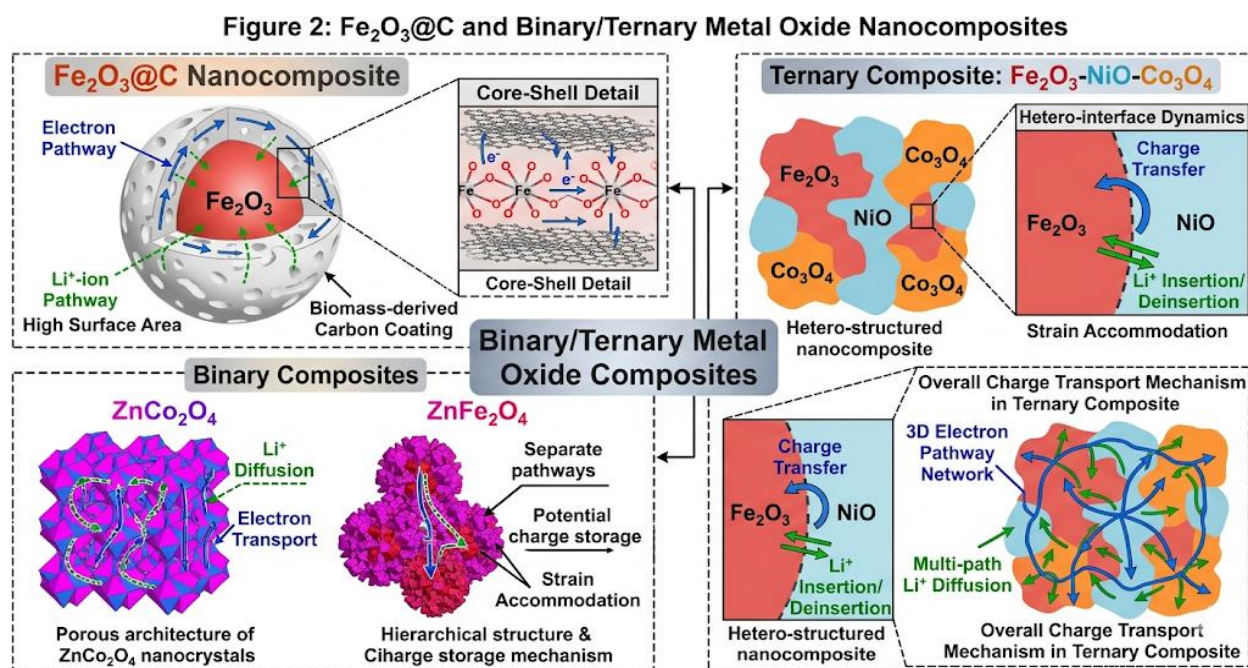
Nickel oxide (NiO) nanoparticles synthesized through green plant-mediated approaches have emerged as auspicious candidates for supercapacitor applications owing to their high specific capacitance and tunable electrochemical properties (Mahmud et al., 2025). NiO nanoparticles were successfully synthesized by utilizing the waste shell powder of *Prunus dulcis* (almond) at 60 degrees C and pH 12 (Patil et al., 2025). The almond shell powder acted as a stabilizing and reducing agent, and although XRD and SEM showed the amorphous nature of the synthesized material, cyclic voltammetry indicated reversible redox behavior (Meena et al., 2024). A one-step green synthesis of NiO nanoparticles using Aloe vera leaf extract yielded remarkable results. The single electrode achieved a specific capacitance of 462 F/g at 1 mV/s and 336 F/g at a current density of 0.76 A/g in 2 M KOH electrolyte (Singh et al., 2024). When applied in a symmetric device (NiO|NiO), it showed a specific capacitance of 239 F/g, an energy density of 47.8 Wh/kg, and a power density of 545 W/kg. This device maintained 89% retention after 2000 cycles at 10 A/g (Kumar & Mishra, 2024).

The synthesis of NiO nanoparticles utilizing Citrus limon leaf extract produced nanocrystalline green NiO (NiO_G) for investigate as an anode material in LIBs. Pure cubic formation was confirmed by XRD, with average crystallite sizes in the order of 20 to 15 nm (Shahab et al., 2025). Integrating graphene oxide (GO) to form a NCG 20 composite resulted in the presence of oxygen vacancies and mixed oxidation states (Ni³⁺/Ni²⁺), which are beneficial for electronic conductivity. Similarly, CuO nanoparticles are synthesized using plant

sources such as *Averrhoa carambola*, *Psidium guajava*, *Catha edulis*, and *Moringa oleifera* (Jeyakumar et al., 2025). The biomolecules facilitate the conversion of the metal precursor into the desired NPs and prevent agglomeration. Biogenic CuO nanoparticles exhibit remarkable charge storage capabilities due to tunable redox properties and high surface area (Rakshit et al., 2025).

6. Iron Oxide and Binary Metal Oxide Systems

Among TMOs, Fe₂O₃ has attracted significant attention due to its high theoretical capacity (~1005 mAh/g), low cost, and natural abundance. It stores Li via a conversion reaction, but its practical application is limited by low conductivity and large volume expansion (Fleischmann et al., 2022). To address these issues, an Fe₂O₃@C composite was developed where Fe₂O₃ nanoparticles were uniformly coated with biomass-derived carbon from bagasse (Zhong, 2020). With an optimized Fe₂O₃:C ratio of 8:2, the electrode delivered a high reversible capacity of 1893 mAh/g after 100 cycles at 0.1 A/g and retained 1553 mAh/g after 350 cycles at 0.5 A/g (Nguyen et al., 2025). Another study utilized seedless pods of *Acacia nilotica* to create Fe₂O₃/C nanoparticles (FeC-2), achieving a specific capacitance of 482.85 F/g compared to 155.71 F/g for FeC-1, demonstrating the influence of plant extract concentration on electrochemical performance (Kiruthika et al., 2022). Figure 2 presents the structural configuration of Fe₂O₃@C composites and bimetallic/ternary TMOs, demonstrating how hybrid architectures enhance conductivity and electrochemical performance.



Binary-metal oxide nanomaterials, such as zinc cobalt (ZnCo₂O₄) and zinc iron (ZnFe₂O₄), demonstrate exceptional properties for catalysis, sensors, and energy storage. These nanoscale composites, typically 1 to 100 nm in size, exhibit distinct physicochemical properties like high surface-to-volume ratios and tunable electronic properties (Matinise, 2025). The green synthesis of these bimetallic oxides involves salt precursors reacting with bioactive compounds from plants like *Moringa oleifera* (Iqbal & Khan, 2021). For instance, biological compounds like L-ascorbic acid undergo oxidation, and the resulting anions attract metal cations to form complexes that are

subsequently annealed. ZnCo₂O₄ exhibits high electrical conductivity and excellent catalytic activity, while ZnFe₂O₄ is characterized by magnetic responsiveness and chemical stability (Clark, 2020).

Ternary nanocomposites like Fe₂O₃-NiO-Co₃O₄ synthesized via an eco-friendly hydrothermal approach using banana peel extract showed a high specific capacity of 2692 mAh/g, significantly surpassing binary compounds. Banana peel extract acts as a green reductant by donating electrons from its polyphenolic (-OH) and carbonyl (C=O) groups to metal ions (Co²⁺, Ni²⁺, Fe³⁺) (Panjaitan et al., 2022).

Table 2: Electrochemical Performance of Green-Synthesized Materials

Material System	Specific Capacity/Capacitance	Cycling Performance	Application
MnO ₂ (Orange Peel)	119 F/g (initial)	100% recovery (500 cycles)	Supercapacitor
NiO (Aloe Vera)	462 F/g	89% retention (2000 cycles)	Supercapacitor
Fe ₂ O ₃ /C (Acacia)	482.85 F/g	-	Supercapacitor
Fe ₂ O ₃ @C (Bagasse)	1893 mAh/g (0.1 A/g)	1553 mAh/g (350 cycles)	LIB Anode
gamma-Fe ₂ O ₃ NPs	1100 mAh/g	Stable 770 mAh/g (high rate)	LIB Anode
SnO ₂ /C (Banana)	1110 mAh/g	660 mAh/g at -20 degrees C	LT LIB Anode
Fe ₂ O ₃ -NiO-Co ₃ O ₄	2692 mAh/g	-	Energy Storage
Co ₃ O ₄ @MnO ₂ @ZnO	1250 F/g (0.5 A/g)	97.25% retention (10k cycles)	Supercapacitor

7. Transition from Lithium-Ion to Sodium-Ion Batteries

Lithium-ion batteries (LIBs) have revolutionized energy storage, but concerns related to the limited availability of lithium have driven interest in sodium-ion batteries (SIBs) as a promising alternative. Sodium is the second-lightest alkali metal, abundant, and significantly cheaper than lithium (Nsude et al., 2025). SIBs share similar architecture and principles with LIBs, making them environmentally benign and cost-effective. However, sodium's larger ionic radius (1.02 angstrom vs. 0.76 angstrom) and heavier atomic mass contribute to slower ion kinetics and lower gravimetric capacity (1165 mAh/g vs. 3829 mAh/g) (Farhan et al., 2025).

Layered transition metal oxides are promising cathodes for SIBs due to their structural similarity to LIB counterparts. Sodium nickel iron manganese oxide (NaNi_{1/3}Fe_{1/3}Mn_{1/3}O₂, or NaNFM) is a high-performance cathode active material (Ruppert et al., 2025). Nickel supplies high capacity and elevates operating voltage; iron increases voltage and structural stability; and manganese minimizes structural distortion (Schumacher, 2025). NaNFM adopts an O₃-type layered structure where sodium ions occupy octahedral sites, facilitating efficient transport. It typically exhibits a first cycle discharge capacity of ~125 mAh/g at 0.1 C and operates between 2.0 V and 4.0 V. Charging beyond 4.0 V can cause irreversible capacity loss (Xiao et al., 2020).

Table 3: Comparison of Sodium-Ion and Lithium-Ion Battery Characteristics

Characteristic	Sodium-Ion Batteries (SIBs)	Lithium-Ion Batteries (LIBs)
Energy Density	150 to 200 Wh/kg	200 to 300 Wh/kg
Nominal Voltage	3.2 to 3.3 V	3.6 to 3.7 V
Availability	Worldwide; ~500x more than Li	Limited reserves
Current Collector	Aluminum for both anode/cathode	Aluminum (cathode), Copper (anode)
Safety/Transport	Can be transported at 0 V	Must be stored with minimum charge
Ionic Radius	1.02 angstrom	0.76 angstrom

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8. Factors Influencing Reproducibility and Scalability

While green synthesis offers numerous benefits, several challenges remain regarding scalability, reproducibility, and the lack of standardized protocols. Plant and vegetable extracts are chemically complex, and limited studies have examined which specific molecules are effectively acting in the synthesis (Ekechukwu et al., 2024). Seasonal variation plays a crucial role, as different seasons alter the metabolites produced by plants and microbes due to changes in temperature and light availability. Geographical variation also affects properties because of differences in soil composition, climate, and altitude, which change the biochemical profile of the source (Badruhisham et al., 2024). Genetic variation within the same species can lead to differences in the concentration of compounds like flavonoids and phenolics, resulting in nanoparticle size and efficiency (Iqbal & Khan, 2021).

Biological synthesis methods utilizing fungi have emerged as viable alternatives offering scalable methods for production. For instance, *Aspergillus niger* and *Aspergillus terreus* act as biological reducing and capping agents for ZnO NPs (Verma & Sidhu, 2024). However, fungal cultures generally grow more slowly than bacterial cultures, requiring longer incubation times and extensive trial and error to optimize conditions. Variations in medium composition, aeration, and light exposure significantly affect the yield and quality, making reproducibility difficult (Verma & Sidhu, 2024).

Scalability scales within green syntheses include reproducibility issues, optimum reaction conditions, and effective supply chains of biopolymers. Strategies to make production even more sustainable on a larger scale include closed-loop systems and recycling residual biomaterials (Rakshit et al., 2025). Integration with artificial intelligence and machine learning is a worthwhile

consideration for future advancements to strengthen the economic viability of green processes (Perumalsamy et al., 2024).

9. Characterization and Advanced Analytical Techniques

Characterization techniques are essential to ensure the precise understanding of nanoparticle properties. Structural, morphological, and chemical analyses are standard (Parveen et al., 2025). X-ray diffraction (XRD) confirms the successful synthesis and crystalline phases, such as the diffraction planes of MoO₃ and rGO in composites (Parveen et al., 2025). Average crystallite size is often determined using the Debye-Scherrer equation (et al., 2022). Scanning Electron Microscopy (SEM) and Field Emission Scanning Electron Microscopy (FESEM) provide morphological analysis, showing shapes like spherical, round-like, or nanoflower structures (Sindhushree et al., 2022).

Fourier-Transform Infrared Spectroscopy (FTIR) identifies phytochemical capping on the nanoparticle surface, verifying synthesis by the appearance of characteristic vibrational peaks, such as the Ni-O stretch at 611.97 cm⁻¹ or Mn-O bonds (Radulescu et al., 2023). UV-Visible spectroscopy assesses surface plasmon resonance and verifies the formation of nanoparticles by measuring absorption peaks, such as the peak at 376 nm for ZnO or 260 nm for MnO₂ (Parveen et al., 2025; et al., 2022). Electrochemical evaluations are typically performed using Cyclic Voltammetry (CV), Galvanostatic Charge-Discharge (GCD), and Electrochemical Impedance Spectroscopy (EIS). EIS analysis in a frequency range of 0.01 to 10⁵ Hz provides insights into charge transfer kinetics and ion diffusion (Kumar & Mishra, 2024).

10. Conclusions

Green synthesis of transition metal oxide nanoparticles represents a sustainable paradigm shift in electrochemical energy storage, harnessing biological agents to produce high-performance materials with minimal environmental footprint. By achieving superior electrochemical metrics high specific capacitance, energy density, and longevity while circumventing

hazardous chemicals, these methods address critical barriers in scaling advanced batteries and supercapacitors for renewable integration and electrification. Despite hurdles in reproducibility and commercialization, ongoing optimizations in precursor selection, hybrid architectures, and process engineering hold immense promise. Ultimately, widespread adoption of biogenic TMOs could significantly reduce global reliance on fossil fuels, mitigate chemical waste, and accelerate the transition to a circular, low-carbon energy economy, underscoring the vital role of interdisciplinary green nanotechnology in future sustainable development.

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