



Electrochemical Performance of Nanostructured Materials for Advanced Energy Storage System

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Peer Review Information

Submission: 04 Jan 2021

Revision: 19 Feb 2021

Acceptance: 03 Mar 2021

Keywords

Nanostructured materials, Electrochemical energy storage, MnO₂/rGO, Supercapacitor, Electrochemical impedance spectroscopy.

Abstract

Nanostructured electrode materials are central to advanced electrochemical energy-storage systems because nanoscale dimensions can shorten ion-diffusion paths, increase electrochemically accessible area, and create interfaces that support rapid charge transfer. This methodology paper presents a reproducible framework for evaluating the electrochemical performance of nanostructured materials using a manganese-dioxide/reduced-graphene-oxide (MnO₂/rGO) hybrid as the principal model electrode, with MnO₂ and rGO controls. The study links hydrothermal synthesis conditions to crystal structure, carbon disorder, morphology, surface area, impedance, rate capability, and cycling behavior. X-ray diffraction, Raman/FTIR spectroscopy, scanning/transmission electron microscopy, and BET analysis are proposed for physicochemical characterization. Electrochemical evaluation uses cyclic voltammetry, galvanostatic charge-discharge, electrochemical impedance spectroscopy, rate-capability testing, and long-term cycling in a controlled aqueous three-electrode configuration, followed by a two-electrode device-level validation. Specific capacitance, equivalent series resistance, charge-transfer resistance, capacitance retention, energy density, and power density are calculated using consistent mass normalization. A multi-criterion comparison is used rather than ranking materials by a single capacitance value. Representative numerical results are included only as methodology-demonstration outputs and are not claimed as unperformed laboratory measurements. The proposed framework can be adapted to graphene hybrids, transition-metal oxides, MXenes, sulfides, and other nanostructured materials intended for advanced energy-storage systems.

Introduction

Advanced batteries and electrochemical capacitors require electrode materials that can simultaneously provide high charge-storage capacity, rapid ion/electron transport, structural stability, and scalable processing. Nanostructuring is attractive because it changes the characteristic transport length and increases the fraction of atoms located at surfaces and interfaces. Aricò et al. [1] established the broad importance of nanostructured electrodes and electrolytes for energy-conversion and storage

technologies, while Bruce, Scrosati, and Tarascon [2] showed how nanoscale design can improve kinetics in rechargeable lithium systems. These benefits are accompanied by challenges such as increased surface reactivity, lower packing density, and more complicated electrode/electrolyte interfaces, so electrochemical performance must be evaluated together with structural stability.

For electrochemical capacitors, material selection and pore/interface engineering are

especially important. Simon and Gogotsi [3] described the major charge-storage mechanisms and materials used in electrochemical capacitors. Graphene became a prominent candidate because its high electrical conductivity and two-dimensional morphology can form fast electron pathways. Stoller et al. [4] demonstrated graphene-based ultracapacitor electrodes, and activated graphene later showed that accessible porosity can strongly increase capacitive performance [9]. These studies also illustrate why nominal surface area alone is insufficient; ion-accessible pore structure, electrolyte wetting, and electrode density must be considered.

Transition-metal oxides add faradaic or pseudocapacitive charge-storage mechanisms. Nanostructured MnO₂ is particularly attractive because manganese is abundant and the oxide can be prepared in multiple phases and morphologies. Subramanian, Zhu, and Wei [5] reported hydrothermal nanostructured MnO₂ for supercapacitor electrodes. Because MnO₂ has modest intrinsic electronic conductivity, hybridization with conductive carbon is a common strategy. Chen et al. [6] deposited MnO₂ on graphene oxide, Wu et al. [7] used MnO₂ nanowire/graphene configurations in asymmetric electrochemical capacitors, and Yu et al. [8] demonstrated scalable solution-processed graphene/MnO₂ textiles. He et al. [11] further showed that three-dimensional graphene/MnO₂ networks can combine a conductive scaffold with a high fraction of redox-active material.

Other nanostructured material families have broadened the design space. Lukatskaya et al. [10] demonstrated cation intercalation and high volumetric capacitance in two-dimensional Ti₃C₂ MXene. Ghidui et al. [12] subsequently reported a conductive two-dimensional titanium-carbide clay with high volumetric capacitance, and Wang et al. [13] demonstrated

pseudocapacitive behavior of MXene nanosheets in high-power sodium-ion hybrid capacitors. Metallic 1T-MoS₂ nanosheets also showed supercapacitor behavior [14]. These systems reinforce a general principle: favorable electrochemical performance emerges when nanostructure, electronic conductivity, ion transport, and reversible redox chemistry are optimized together.

As the field matured, greater emphasis was placed on mechanistic understanding and realistic reporting. Salanne et al. [15] reviewed charge-storage mechanisms in carbon- and oxide-based supercapacitors and emphasized the role of ion confinement and interfacial structure. Pomerantseva et al. [16] described how nanomaterials can enable future batteries and capacitors but also identified issues of scalability, stability, and electrode density. The 2021 review by VahidMohammadi, Rosen, and Gogotsi [17] summarized the expanding MXene family and its relevance to electrochemical applications. On this basis, the present study defines a methodology that connects synthesis, characterization, and electrochemical performance using a MnO₂/rGO hybrid as a controllable model while maintaining principles applicable to broader nanostructured energy-storage materials.

Literature Review

Selected literature up to the requested 2021 cutoff is summarized in Table 1. The papers were chosen to represent fundamental nanostructuring concepts, graphene and MnO₂ hybridization, two-dimensional energy-storage materials, and modern mechanistic perspectives. Together they justify evaluating not only capacitance but also rate response, resistance, cycling stability, volumetric/gravimetric trade-offs, and device-level behavior.

Table 1. Research Basis for Nanostructured Supercapacitor Electrode Methodology

Study	Material / concept	Key finding	Relevance methodology to	Ref.
Aricò et al. (2005)	Nanostructured energy materials	Nanoscale structures improve transport/interfacial behavior	Structure-performance framework	[1]
Bruce et al. (2008)	Battery nanomaterials	Nanostructuring changes storage kinetics and interfaces	Account for transport and stability	[2]

Simon & Gogotsi (2008)	Electrochemical capacitors	Defined EDLC/pseudocapacitive material principles	Select appropriate electrochemical metrics	[3]
Stoller et al. (2008)	Graphene	Graphene electrodes enable high capacitive response	Conductive-carbon reference	[4]
Subramanian et al. (2006)	Nanostructured MnO ₂	Hydrothermal MnO ₂ for supercapacitors	Oxide synthesis basis	[5]
Chen et al. (2010)	GO-MnO ₂	Facile oxide deposition on graphene oxide	Hybrid nucleation strategy	[6]
Wu et al. (2010)	MnO ₂ /graphene	Asymmetric capacitor with improved energy response	Device-level validation	[7]
Yu et al. (2011)	Graphene/MnO ₂ textile	Solution-processed hybrid electrodes	Scalable hybrid processing	[8]
Zhu et al. (2011)	Activated graphene	Accessible porosity enhanced capacitance	BET must be linked to ion access	[9]
Lukatskaya et al. (2013)	Ti ₃ C ₂ MXene	High volumetric capacitance via intercalation	Importance of 2D ion pathways	[10]
He et al. (2013)	3D graphene/MnO ₂	Conductive 3D network with oxide loading	Interface/connectivity design	[11]
Ghidiu et al. (2014)	Ti ₃ C ₂ Tx clay	High volumetric capacitance in MXene films	Include volumetric considerations	[12]
Wang et al. (2015)	MXene pseudocapacitance	High-rate Na-ion hybrid storage	Rate-capability benchmark concept	[13]
Acerce et al. (2015)	Metallic 1T-MoS ₂	2D metallic phase showed capacitor behavior	Alternative nanostructure comparison	[14]
Salanne et al. (2016)	Storage mechanisms	Connected structure and ion confinement to capacitance	Mechanistic interpretation	[15]
Pomerantseva et al. (2019)	Future nanomaterials	Highlighted opportunities and practical constraints	Scalability/stability discussion	[16]
VahidMohammadi et al. (2021)	MXenes	Comprehensive 2D carbide/nitride perspective	2021 state-of-the-art context	[17]

The literature indicates that material comparisons can become misleading if different active-mass loadings, voltage windows, cell configurations, or calculation conventions are used. The present methodology therefore keeps electrode area, active loading, electrolyte, potential window, current-density sequence, and data-analysis equations constant for the three comparative materials. The central hypothesis is that an intermediate MnO₂ loading on a continuous rGO network will provide a better compromise between pseudocapacitive charge

storage and electronic/ionic transport than either MnO₂ or rGO alone.

Methodology

A controlled comparative experimental design is proposed with three principal samples: rGO (carbon control), hydrothermal MnO₂ (oxide control), and MnO₂/rGO (hybrid test material). The study is divided into synthesis, physicochemical characterization, electrode fabrication, three-electrode electrochemical screening, two-electrode device validation, and

statistical comparison. Figure 1 presents the workflow.



Figure 1. Proposed synthesis-to-electrochemical-performance workflow for nanostructured energy-storage electrodes.

1. Experimental Variables and Sample Design

The independent variable is nanostructure/composition. The rGO control represents a predominantly double-layer conductive material, the MnO₂ control represents a pseudocapacitive oxide, and the MnO₂/rGO sample represents an interface-engineered hybrid. To examine composition sensitivity, the hybrid can be synthesized at low, medium, and high nominal MnO₂ loadings while all other conditions are held constant. Dependent variables include BET surface area, crystallite/particle morphology, specific capacitance, equivalent series resistance, charge-transfer resistance, rate retention, coulombic efficiency, and cycle-life retention. At least three independently prepared electrodes per composition are recommended.

Step-by-Step Synthesis Procedure

Step 1: Disperse graphene oxide in deionized water using controlled stirring and sonication until a stable, visibly uniform suspension is obtained.

Step 2: Prepare the manganese precursor solution separately and add it slowly to the graphene-oxide dispersion under continuous mixing so manganese species distribute across oxygen-containing nucleation sites.

Step 3: Adjust precursor-to-GO ratio to generate low-, medium-, and high-MnO₂-loading hybrids. Maintain identical total volume, pH strategy, mixing time, and precursor purity.

Step 4: Transfer the mixture to a sealed hydrothermal reactor and apply a moderate hydrothermal treatment consistent with MnO₂ nanostructure formation reported in [5]. The exact temperature/time should be optimized in preliminary runs and then fixed for comparative testing.

Step 5: Allow natural cooling, separate the product, and wash repeatedly with deionized

water until soluble precursor residues are minimized.

Step 6: Dry the material under controlled low-temperature conditions. Apply mild thermal or chemical reduction only when needed to convert GO toward rGO while avoiding unwanted MnO₂ phase transformation.

Step 7: Prepare pure rGO and pure MnO₂ references with matched processing steps wherever practical. Store powders in sealed containers and assign batch codes for traceability.

Step 8: Record precursor masses, solution volumes, reaction temperature, time, drying condition, final yield, and nominal oxide fraction for every batch so structure-performance trends can be reproduced.

2. Structural and Surface Characterization

X-ray diffraction is used to identify MnO₂ phase formation and to detect broad carbon-related features. Raman spectroscopy evaluates the carbon D and G bands and confirms oxide-related vibrational signatures; the ID/IG ratio is tracked as a qualitative indicator of carbon disorder after processing. FTIR can support identification of residual oxygen-containing groups and metal-oxygen bonding. SEM is used to determine whether the oxide forms nanoparticles, nanorods/nanosheets, or agglomerates on the rGO network. TEM is recommended for direct observation of nanoscale interface contact. BET nitrogen adsorption/desorption provides specific surface area and pore-size distribution. These data are interpreted together because high surface area is useful only when electrolyte ions can access the relevant pores and the particles remain electronically connected.

3. Electrode Fabrication and Cell Configuration

A conventional slurry route is used for consistent mass normalization. The active material,

conductive carbon, and binder are mixed at an approximately 80:10:10 mass ratio unless a binder-free current collector is specifically studied. The slurry is coated onto cleaned nickel foam, stainless-steel mesh, or carbon cloth, dried, and lightly pressed. Active mass loading is kept within a narrow range, for example approximately 1.5-2.5 mg cm⁻², across all samples. Initial intrinsic performance is measured in a three-electrode aqueous cell using an inert counter electrode and a stable reference electrode. A neutral electrolyte such as 1 M Na₂SO₄ is selected to minimize corrosive side reactions. The usable potential window is established by preliminary CV so that water decomposition is avoided.

4. Electrochemical Measurement Protocol

Cyclic voltammetry is recorded at 5, 10, 20, 50, and 100 mV s⁻¹. Retention of curve shape with

increasing scan rate is used as a qualitative indicator of fast charge transport. Galvanostatic charge-discharge is performed at increasing gravimetric current densities, for example 0.5, 1, 2, 5, and 10 A g⁻¹. Electrochemical impedance spectroscopy is collected from approximately 100 kHz to 10 mHz using a small AC perturbation at a defined equilibrium potential. Equivalent series resistance is estimated from the high-frequency intercept, while the semicircle region is interpreted in relation to charge-transfer resistance. Long-term cycling is conducted for at least 5,000 cycles at a moderate current density. For the best electrode, a two-electrode asymmetric or symmetric device is assembled and evaluated separately to avoid mixing three-electrode and practical-device performance metrics.

Table 2. Summary of Electrochemical Tests and Evaluation Parameters

Test	Condition	Primary metric	Interpretation
CV	5-100 mV s ⁻¹	Current response / integrated charge	Kinetics and reversibility
GCD	0.5-10 A g ⁻¹	Specific capacitance, iR drop	Capacity and rate capability
EIS	100 kHz-10 mHz	Rs, Rct, diffusion trend	Electronic/interfacial transport
Cycling	>=5,000 cycles	Capacitance retention	Structural/electrochemical stability
Device test	Two-electrode configuration	Energy and power density	Practical system validation
Post-cycle analysis	SEM/Raman where available	Morphology/structure change	Failure-mechanism evidence

5. Calculation and Data Analysis

For a single electrode evaluated by galvanostatic discharge, gravimetric specific capacitance is calculated as $C_{sp} = I\Delta t / (m\Delta V)$, where I is discharge current, Δt is discharge time, m is the active-material mass on the working electrode, and ΔV is the effective discharge interval after excluding the instantaneous iR drop. Rate retention is C_{sp} at the highest selected current density divided by C_{sp} at the lowest selected current density. Cycling retention is $C_n/C_0 \times 100\%$. Coulombic efficiency is estimated from the ratio of discharge to charge time under matched current. For a properly balanced two-electrode device, energy density is calculated from $E = 0.5 C_{device}(\Delta V)^2/3.6$ and power density from $P = 3600E/\Delta t$. Three-electrode capacitance must not be inserted directly into device-level energy calculations because the mass basis and voltage definition differ.

For statistical analysis, replicate electrode data are reported as mean $\pm$ standard deviation. One-way ANOVA can be applied to compare sample groups when assumptions are satisfied; otherwise, a suitable non-parametric test is preferred. The optimum material is selected through multi-criterion ranking. A sample with the highest low-rate capacitance is not considered optimal if it shows poor rate retention, large R_{ct} , unstable cycling, or unrealistic active-mass loading.

Results

The numerical values in this section are illustrative, literature-informed methodology outputs used to demonstrate how the proposed experiment should be reported. They are not presented as measurements from laboratory work performed for this paper. A final experimental article should replace these values

with measured means, standard deviations, fitted impedance parameters, and raw electrochemical curves.

Table 3. Comparative Performance Parameters of rGO, MnO₂, and MnO₂/rGO Samples

Sample	BET area (m ² g ⁻¹)	Csp at 1 A g ⁻¹ (F g ⁻¹)	Csp at 10 A g ⁻¹ (F g ⁻¹)	Rate retention (%)	Rct (ohm)	5000-cycle retention (%)
rGO	225	160	118	74	2.6	95
MnO ₂	132	245	137	56	4.5	87
MnO ₂ /rGO-L	188	305	220	72	2.1	92
MnO ₂ /rGO-M	201	392	302	77	1.5	96
MnO ₂ /rGO-H	169	351	226	64	2.4	91

The illustrative trend supports the proposed transport/storage balance. rGO shows relatively low charge-transfer resistance and excellent cycling retention but lower overall capacitance because charge storage is dominated by double-layer processes. MnO₂ provides larger low-rate capacitance through pseudocapacitive redox reactions but suffers greater resistance and a sharper loss at high current density. The hybrid samples outperform the individual controls because rGO provides an electronically conductive framework and MnO₂ supplies additional redox-active sites, consistent with the interfacial strategies reported in [6-8,11].

The medium-loading hybrid is the illustrative optimum. Its 392 F g⁻¹ low-rate capacitance is accompanied by 77% rate retention, an Rct of 1.5 ohm, and 96% retention after 5,000 cycles. Increasing MnO₂ beyond this point does not continue the improvement because thicker or more aggregated oxide domains can obstruct conductive pathways and reduce electrolyte-accessible porosity. This interpretation is consistent with the broader literature showing

that nanostructure must maintain both ion access and electronic continuity [1,3,15].

EIS is especially important for validating the mechanism. If the optimized hybrid displays a smaller high-frequency semicircle than pure MnO₂, the improvement can be associated with reduced interfacial charge-transfer resistance rather than only a larger active mass. A more nearly vertical low-frequency response would additionally support improved capacitive ion transport. Post-cycle SEM/Raman should then be used to determine whether the hybrid network suppresses oxide agglomeration, detachment, or structural degradation. This combined interpretation avoids relying on a single capacitance number.

Illustrative Two-Electrode Device Validation

After three-electrode screening, the medium-loading hybrid is selected for device-level validation. The two-electrode test is treated as a separate stage because practical energy and power densities depend on the full cell voltage, balancing of positive/negative electrodes, total active mass, and electrolyte stability.

Table 3. Summary of Device-Level Energy Storage Performance and Stability

Device metric	Illustrative value	Evaluation condition	Acceptance interpretation	Comment
Device capacitance	78 F g ⁻¹	1 A g ⁻¹	Stable full-cell response	Total active-mass basis
Energy density	24.4 Wh kg ⁻¹	Defined device window	Useful hybrid-capacitor range	Calculated from device capacitance
Power density	0.82 kW kg ⁻¹	Moderate discharge rate	High-power capability retained	Report with discharge time
High-power retention	61%	10 A g ⁻¹	Acceptable rate response	Compared with low-rate device value

Cycle retention	93%	5,000 cycles	Good device stability	Must be experimentally verified
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The device-level analysis also illustrates why advanced nanomaterials should be discussed in terms of practical trade-offs. MXenes can provide exceptional volumetric capacitance [10,12,13], activated graphene can provide rapid double-layer storage [9], and metallic 1T-MoS₂ offers another two-dimensional pseudocapacitive route [14]. Nevertheless, material selection for an advanced energy-storage system must also consider density, stability, scalable synthesis, electrolyte compatibility, cost, and safety. The wider assessments in [16] and [17] reinforce that nanomaterials can enable major performance gains but do not eliminate system-level engineering constraints.

Conclusion and Discussion

This methodology paper develops a structured approach for evaluating the electrochemical performance of nanostructured materials for advanced energy-storage systems. A MnO₂/rGO hybrid is used as a representative material because it combines a conductive two-dimensional carbon network with a redox-active metal oxide. The methodology integrates controlled synthesis, XRD/Raman/FTIR analysis, SEM/TEM morphology, BET surface characterization, electrochemical screening by CV/GCD/EIS, rate-capability testing, long-term cycling, and separate two-electrode device validation. This progression reflects the literature evolution from basic nanostructure concepts [1,2] and graphene/oxide electrodes [4-8,11] to advanced two-dimensional and pseudocapacitive materials [10,12-14,17]. The central methodological conclusion is that electrochemical performance should be treated as a multi-variable outcome. High capacitance is valuable only when supported by low resistance, fast-rate response, reproducible active-mass loading, cycling stability, and a credible full-device voltage window. The illustrative data show an expected optimum at intermediate MnO₂ loading: insufficient oxide limits redox capacity, whereas excessive oxide can obstruct conductive pathways and reduce utilization. Such a structure-performance relationship is consistent with modern mechanistic understanding of confined ions, interfacial charge storage, and transport [3,15]. The methodology has several limitations that should be acknowledged. Three-electrode measurements tend to overstate performance relative to full devices; aqueous electrolytes restrict cell voltage; surface-area values do not

directly quantify electrochemically active surface; and nanomaterial performance can deteriorate when active-mass loading is increased toward practical levels. Accordingly, future work should include higher areal mass loadings, volumetric capacitance, self-discharge, leakage current, temperature dependence, post-mortem microscopy, and long-duration two-electrode testing. The broader perspective on nanomaterials for future energy storage [16] and the 2021 MXene review [17] also suggest comparing the proposed MnO₂/rGO system with alternative two-dimensional, oxide, sulfide, and hybrid electrodes under identical device-level protocols.

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