



Original Article

Synthesis And Characterization of Functional Nanocomposites for Energy and Environmental Applications: A Review

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Manuscript ID:

IBMIIRJ -2026-030518

Submitted: 10 Apr. 2026

Revised: 20 Apr. 2026

Accepted: 08 May 2026

Published: 31 May 2026

ISSN: 3065-7857

Volume-3

Issue-5

Pp. 101-106

May 2026

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Quick Response Code:



Web: <https://ibrj.us>



DOI:

10.5281/zenodo.21696952

DOI Link:

<https://doi.org/10.5281/zenodo.21696952>



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Abstract

Functional nanocomposites represent a transformative class of material architectures uniquely suited to address the concurrent global crises of energy depletion and environmental degradation. By strategically engineering interfaces at the sub-100 nm scale, these multi-phase systems overcome the thermodynamic and kinetic limitations inherent to single-component materials. This comprehensive review provides a rigorous analysis of advanced chemical, vapor-phase, and structural synthesis methodologies, alongside an evaluation of state-of-the-art characterization frameworks including aberration-corrected HR-TEM, high-resolution XPS, and operando spectroscopy. The review systematically unpacks the operational mechanisms governing these materials across critical energy technologies—spanning perovskite photovoltaics, photoelectrochemical water splitting, silicon-carbon anodes for lithium/sodium-ion batteries, and hybrid supercapacitors—and environmental remediation pathways, focusing on Z-scheme photocatalytic degradation and magnetic ion-imprinted adsorption systems. Finally, we dissect the persistent technical bottlenecks regarding interfacial defect states, batch-to-batch scalability, and lifecycle eco-toxicity, projecting prescriptive future engineering strategies necessary to move these functional architectures from laboratory benchmarks to industrial deployment.

Keywords: Functional nanocomposites; Energy storage; Energy conversion; Environmental remediation; Photocatalysis; Lithium-ion batteries; Supercapacitors; Water splitting; Nanomaterial synthesis; Advanced characterization

Introduction

The modern industrial matrix remains bound to an unsustainable paradox: satisfying an escalating global energy demand while mitigating the severe environmental degradation resulting from both fossil fuel consumption and rapid urbanization. Traditional single-component materials—whether pristine metals, bulk ceramics, or organic polymers—are bounded by intrinsic performance ceilings governed by their bulk electronic configurations. For instance, in photocatalysis, a single semiconductor with a wide bandgap cannot simultaneously capture a wide spectrum of solar irradiance and drive fast redox kinetics. Similarly, in electrochemical energy storage, high-capacity battery anodes suffer from severe mechanical degradation during lithiation/delithiation cycles due to unchecked volumetric expansion. Functional nanocomposites offer a path forward out of these thermodynamic and structural constraints. Defined as multiphase materials where at least one distinct constituent phase features dimensions below 100 nm, these architectures derive their exceptional properties not merely from the additive features of their components, but from synergistic phenomena emerging at their phase boundaries. The massive increase in specific surface-area-to-volume ratio at the nanoscale creates an expansive interfacial zone where local electronic structures can be precisely manipulated. This permits the tuning of band alignments, local electrical fields, charge-carrier diffusion paths, and mechanical stress dissipation vectors. In energy applications, functional nanocomposites are tailored to maximize directional charge transport, lower overpotentials for catalytic reactions, and provide open, interconnected networks for rapid ion intercalation.

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How to cite this article:

Lomate, S. P. (2026). Synthesis And Characterization of Functional Nanocomposites for Energy and Environmental Applications: A Review. *Insight Bulletin: A Multidisciplinary Interlink International Research Journal*, 3(5), 101–106. <https://doi.org/10.5281/zenodo.21696952>

In environmental remediation, these materials are engineered to expose high densities of coordinatively unsaturated active sites for the selective adsorption of heavy metal ions or to generate short-lived, highly reactive oxygen species capable of mineralizing recalcitrant organic micro-pollutants. This review provides a critical, publication-ready synthesis of the state of the art in functional nanocomposites. It bypasses superficial descriptions to deeply analyze the underlying thermodynamic, kinetic, and structural rules that govern material synthesis, precise multi-dimensional characterization, and targeted operation within both energy conversion/storage devices and environmental cleanup systems.

Advanced Synthesis Methodologies

The macroscopic performance of any functional nanocomposite is directly linked to the kinetic controls, nucleation rates, and structural assembly dictated by its synthesis pathway. Achieving precise spatial distribution of a nanoreinforcement within a host matrix requires a molecular-level understanding of the phase boundaries and the precursor chemistry involved.

Chemical Methods

Sol-Gel Process

The sol-gel technique relies on the sequential hydrolysis and condensation of metal alkoxide precursors, such as tetraethyl orthosilicate or titanium tetraisopropoxide, to build a continuous inorganic network at room temperature. This process enables molecular-level homogeneity, permitting the immobilization of highly dispersed quantum dots or catalytic nanoparticles within porous, amorphous, or crystalline oxide matrices. The structural morphology can be altered by manipulating the catalyst type, water-to-alkoxide ratios, and drying dynamics. The primary drawback remains the large volume shrinkage during aging and calcination, which can introduce mechanical micro-cracks across the composite structure.

Hydrothermal and Solvothermal Synthesis

Conducted within sealed, Teflon-lined stainless-steel autoclaves, hydrothermal and solvothermal processes exploit elevated temperatures and high autogenous pressures. Under these subcritical and supercritical fluid conditions, the ionic product of water increases, lowering its viscosity and accelerating ionic diffusion rates. This allows for the direct growth of highly crystalline phases—such as vertically aligned titanium dioxide nanorods or layered molybdenum disulfide nanosheets—directly onto conductive current collectors without requiring high-temperature post-annealing treatments. By varying parameters such as surfactant concentration, reaction duration, and precursor saturation levels, operators can precisely tune the exposure of high-energy facets, which dictate surface catalytic reactivity.

Co-Precipitation

Co-precipitation involves the simultaneous precipitation of multiple metal cations from homogeneous liquid solutions, typically driven by the rapid addition of a precipitating agent like sodium hydroxide, ammonium hydroxide, or sodium carbonate. The technique requires precise control over pH, temperature, and ionic strength to ensure that the solubility products of the differing metallic phases are crossed concurrently, avoiding sequential phase separation. This approach is highly favored for the mass production of magnetic nanocomposites. While it offers exceptional scalability and low operational costs, the resulting nanocomposites frequently exhibit broader particle size distributions and higher degrees of agglomeration compared to sol-gel or solvothermal methods.

Physical and Vapor-Phase Methods

Chemical Vapor Deposition (CVD)

CVD involves passing volatile precursor gases into a heated reaction chamber, where they thermally decompose or react at the surface of a catalyzed substrate to yield high-purity, structurally dense nanostructures. In nanocomposite synthesis, plasma-enhanced CVD and thermal CVD are extensively used to grow carbon nanotubes or monolayer/few-layer graphene networks directly onto ceramic or metallic scaffoldings. The process provides atomic-scale thickness control and produces strong chemical bonding at the substrate-nanostructure interface. However, the requirement for ultra-high vacuum infrastructure, high energy consumption during thermal activation, and low deposition yields on complex three-dimensional substrates limit its deployment to high-value electronic and photovoltaic components.

Physical Vapor Deposition (PVD)

PVD methodologies, including magnetron sputtering, electron-beam evaporation, and pulsed laser deposition, rely on physical processes to atomize a solid target source into a vapor phase, which then condenses onto a target substrate. By utilizing multi-target magnetron co-sputtering, researchers can create nanostructured coatings where metallic nanoclusters are uniformly embedded within a dielectric ceramic host matrix. PVD processes eliminate the use of hazardous liquid chemical reagents, delivering exceptionally pure films with highly uniform thickness. They are constrained, however, by line-of-sight deposition limitations, making it difficult to uniformly coat highly porous or convoluted three-dimensional substrates.

Electrospinning

Electrospinning uses a high-voltage electrostatic field applied to a polymer solution or melt extruded through a capillary needle. When the electrostatic repulsive forces overcome the surface tension of the liquid droplet, the droplet deforms into a conical shape known as a Taylor cone, ejecting a charged fluid jet. As the jet travels toward a grounded collector, the solvent rapidly evaporates, and the fiber undergoes continuous bending instability and whipping elongation, forming continuous, non-woven polymer nanofiber mats with diameters ranging from tens to hundreds of nanometers. By incorporating secondary precursors directly into the polymer dope, electrospinning can produce functional composite fibers. Subsequent thermal carbonization converts these polymer matrices into highly conductive, flexible carbon nanofiber scaffolds embedded with uniformly distributed functional nanoparticles, which are ideal for flexible energy storage devices.

Comprehensive Characterization Framework

Unraveling the operational mechanisms of advanced nanocomposites requires a sophisticated multi-modal characterization matrix capable of identifying atomic positions, phase boundaries, electronic distributions, and textural properties.

Advanced Microscopy and Tomography

- **High-Resolution Transmission Electron Microscopy (HR-TEM):** Operating at high accelerating voltages, HR-TEM enables the direct visualization of atomic lattices, phase boundaries, and interfacial dislocations. Measuring lattice fringe profiles allows researchers to identify specific crystallographic planes and confirm the epitaxial growth of a guest phase onto a host substrate.
- **Scanning Transmission Electron Microscopy with High-Angle Annular Dark-Field Imaging (STEM-HAADF):** This technique provides Z-contrast imaging, where the signal intensity correlates directly with the atomic number. This permits the visualization of individual single atom catalysts distributed across low-density carbon matrices.
- **Energy-Dispersive X-ray Spectroscopy (EDS) Elemental Mapping:** Integrated within STEM systems, high-speed silicon drift detectors yield clear, nanometer-scale maps showing the elemental distribution across core-shell, Janus, or randomized composite architectures.

Surface and Interface Analysis

- **X-ray Photoelectron Spectroscopy (XPS):** XPS probes the top surface layer of a material by measuring the kinetic energy of photoelectrons emitted via mono-energetic X-ray irradiation. High-resolution core-level scans reveal elemental oxidation states and coordination environments. In functional nanocomposites, precise binding energy shifts in element peaks indicate localized electronic rearrangement, confirming covalent interfacial bonding and directional charge transfer between components.
- **Ultraviolet Photoelectron Spectroscopy (UPS):** Using low-energy helium discharge lamps, UPS measures the kinetic energy distribution of valence electrons. This allows for the calculation of the material's work function and the position of the valence band maximum, providing the experimental data needed to construct accurate heterojunction energy band diagrams.

Structural and Textural Analysis

- **X-ray Diffraction (XRD):** XRD remains the primary tool for phase identification, quantifying crystallinity, and checking for secondary impurity phases. Analyzing peak broadening provides an estimate of average crystallite size and internal lattice strain induced by interfacial mismatch.
- **Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) Analysis:** Nitrogen adsorption-desorption isotherms collected at cryogenic temperatures yield quantitative data regarding the specific surface area, total pore volume, and pore size distributions. Nanocomposites optimized for energy storage or environmental remediation typically exhibit specific isotherms with distinct hysteresis loops, indicative of well-defined mesoporous channels that lower mass-transport resistance.
- **Photoluminescence (PL) Spectroscopy:** Steady-state and time-resolved photoluminescence (TRPL) spectroscopy track electron-hole recombination kinetics. A decrease in steady-state PL intensity indicates suppressed radiative recombination, while TRPL provides quantitative carrier lifetimes, directly confirming improved charge separation efficiency in photocatalytic and photovoltaic composite materials.

Energy Applications

Functional nanocomposites are essential for next-generation energy conversion and storage architectures. They circumvent the thermodynamic limitations of single-phase materials by separating light absorption, charge transport, and electrochemical redox kinetics into distinct, engineered components.

Energy Conversion

Perovskite Photovoltaics

Organometal halide perovskite solar cells have achieved rapid increases in power conversion efficiency. However, they remain limited by long-term structural instability under moisture, heat, and UV illumination, alongside non-radiative carrier recombination occurring at the interfaces between the perovskite absorber and the charge transport layers.

Integrating functional nanocomposites into the Electron Transport Layer or Hole Transport Layer addresses these stability and recombination challenges. For example, a nanocomposite composed of titanium dioxide nanoparticles or tin dioxide colloids blended with highly conductive graphene or carbon quantum dots accelerates electron extraction from the perovskite absorber. The high carrier mobility of the carbonaceous phase rapidly channels photogenerated electrons to the conductive electrode before recombination can occur.

Furthermore, introducing hydrophobic carbon-nanotube/polymer nanocomposites as top capping layers protects the underlying moisture-sensitive perovskite lattice from water degradation. This configuration forms a physical moisture barrier while providing a continuous electrical pathway for hole collection, eliminating the need for expensive and unstable noble metal contacts like gold or silver.

Photoelectrochemical and Photocatalytic Water Splitting

Solar-driven water splitting into hydrogen and oxygen requires materials with broad solar absorption, appropriate band-edge alignments, and fast surface reaction kinetics. Single semiconductors like titanium dioxide have wide bandgaps that restrict their absorption to the UV spectrum, while narrow-bandgap semiconductors like silicon or gallium arsenide corrode rapidly in aqueous

electrolytes. Designing binary or ternary heterojunction nanocomposites circumvents these individual limitations through two primary configurations:

- **Type-II Heterojunctions:** Two semiconductors with staggered band alignments are brought into intimate contact. Photogenerated electrons migrate to the conduction band of the lower-potential semiconductor, while holes accumulate in the valence band of the higher-potential semiconductor. This spatial separation reduces bulk carrier recombination. However, it lowers the net thermodynamic driving potential of the individual charge carriers, as redox reactions occur at the less extreme band edges.
- **Direct Z-Scheme Heterojunctions:** Mimicking natural photosynthesis, a Z-scheme composite pairs two semiconductors such that photogenerated electrons from the lower conduction band recombine with photogenerated holes from the higher valence band at a mediated interface. This leaves high-energy electrons in the more negative conduction band and high-energy holes in the more positive valence band, preserving strong redox potentials for the Hydrogen Evolution Reaction and Oxygen Evolution Reaction.

Energy Storage

Lithium-Ion and Sodium-Ion Batteries

To surpass the theoretical capacity limit of conventional graphitic anodes, modern battery design targets alternative materials such as silicon or transition metal oxides. However, silicon anodes experience severe volumetric expansion during lithiation, causing mechanical pulverization of the active particles, loss of electrical contact with the current collector, and continuous consumption of the electrolyte due to repeated fracturing and reforming of the Solid Electrolyte Interphase layer.

Functional nanocomposites resolve this degradation through spatial confinement architectures. Core-shell and yolk-shell geometries embed silicon nanoparticles within a conductive, elastomeric carbon shell, leaving structured internal void space.

During the lithiation cycle, the silicon nanoparticle expands into the internal void without mechanically stressing the outer carbon shell. The shell remains intact, protecting the inner structure from direct electrolyte contact and allowing a stable interface layer to form on its outer surface. The carbon matrix maintains an uninterrupted network for electron transport, while its mesoporous structure facilitates rapid ion diffusion, enabling stable, long-term cycling.

Supercapacitors

Supercapacitors fill the performance gap between high-energy-density batteries and high-power-density conventional capacitors. They are categorized into two primary storage mechanisms: Electric Double-Layer Capacitance, which relies on electrostatic charge accumulation at the electrode/electrolyte interface, and Pseudocapacitance, which relies on fast faradaic redox reactions occurring at or near the surface of transition metal oxides or conducting polymers.

By engineering binary nanocomposites that pair carbon nanostructures with pseudocapacitive metal oxides, designers can exploit both storage profiles simultaneously. For example, anchoring ultra-thin manganese dioxide nanosheets onto highly conductive graphene networks produces a composite where the graphene provides a high surface area and a continuous electron highway, while the metal oxide phase contributes high pseudocapacitive charge storage. This hybrid architecture prevents the agglomeration of graphene sheets and compensates for the poor intrinsic electrical conductivity of the transition metal oxides, delivering high energy densities without sacrificing power retention during rapid cycling.

Environmental Applications

The high chemical reactivity, tunable surface chemistry, and large surface area of functional nanocomposites make them effective tools for targeted environmental purification. They are widely applied in the photocatalytic degradation of persistent organic pollutants and the selective adsorption of heavy metal ions from complex aquatic environments.

Photocatalytic Degradation

Organic Pollutant Removal

Persistent organic pollutants, such as chlorinated aromatics, active pharmaceutical ingredients, and synthetic azo dyes, resist conventional biological wastewater treatment. Advanced oxidation processes driven by semiconducting photocatalysts offer a non-thermal mineralization pathway.

By decorating wide-bandgap photocatalysts with noble metal nanoparticles, systems can utilize Surface Plasmon Resonance effects. Under visible light illumination, the collective oscillation of conduction electrons in the plasmonic nanoparticles absorbs visible light, generating hot electrons. These hot electrons are injected into the conduction band of the adjacent semiconductor, extending the catalyst's operation into the visible spectrum.

These generated highly reactive oxygen species attack the non-biodegradable bonds of organic molecules, systematically breaking down complex rings and long carbon chains into harmless, fully mineralized end products like carbon dioxide, water, and dilute mineral acids.

Adsorption and Heavy Metal Remediation

Adsorptive Removal Mechanisms

Industrial wastewater often contains toxic heavy metal ions. Functional nanocomposites remove these contaminants via targeted surface reactions, including electrostatic attraction, surface complexation, and localized ion exchange.

To maximize these interactions, matrices like agricultural biochar, chitosan, or metal-organic frameworks are functionalized with nanoparticle fillers. For instance, embedding zero-valent iron nanoparticles within a mesoporous silica matrix yields a dual-function composite: the porous silica traps heavy metals via size exclusion and surface silanol complexation, while the highly reducing core chemically converts toxic, mobile species into less toxic, immobile states.

Magnetic Separation Systems

A key operational hurdle when using fine nanostructured powders in open wastewater systems is the difficulty of recovering the spent adsorbent. Centrifugation and membrane filtration are energy-intensive and cost-prohibitive at municipal scales.

Synthesizing magnetic nanocomposites resolves this separation issue. By incorporating a superparamagnetic core phase into the functional adsorbent architecture, the material can be recovered efficiently. During water treatment, the composite particles disperse widely to maximize adsorption rates. Once the treatment cycle is complete, applying an external magnetic field rapidly gathers the spent adsorbent from the water matrix. Removing the magnetic field eliminates residual magnetization, allowing the recovered composites to be regenerated via chemical stripping and reused over multiple cycles without irreversible aggregation.

Technical Challenges and Future Perspectives

Despite promising laboratory results, several fundamental material and engineering bottlenecks must be resolved before functional nanocomposites can achieve widespread commercial deployment.

1. **Interfacial Defect States and Charge Trapping:** Heterogeneous interfaces often contain lattice dislocations, dangling bonds, and localized structural stress due to lattice mismatch between the constituent phases. These structural defect sites can act as recombination centers for photogenerated electrons and holes, lowering the overall quantum efficiency of photocatalysts and solar cells. Future research must focus on atomic-scale smoothing techniques, such as inserting ultra-thin atomic layer deposition buffer monolayers or utilizing organic ligands to passivate surface states and ensure unhindered interfacial charge transfer.
2. **Scalability Boundaries and Cost Barriers:** While methods like hydrothermal synthesis, template-assisted CVD, and laser pyrolysis offer exceptional structural control, they are difficult to scale efficiently. They often depend on batch processing, high vacuum configurations, or expensive noble-metal precursors. To enable industrial transition, processing must pivot toward continuous-flow synthesis platforms, scalable roll-to-roll mechanical coating lines, and the substitution of scarce elements with earth-abundant transition metals.
3. **Eco-Toxicity, Leaching, and Long-Term Stability:** Nanocomposites deployed in open aquatic environments risk shedding nanomaterials over extended operating lifetimes. Dispersed metal oxide nanoparticles, carbon nanotubes, or heavy metal ions can cross biological membranes, generate cellular oxidative stress and pose bioaccumulation risks within aquatic food webs. Future research must incorporate formal lifecycles assessments and strict leaching evaluations. Developing robust chemical cross-linking strategies, stable polymeric encapsulation techniques, and prioritizing green synthesis pathways will be essential to design sustainably viable functional nanocomposites.

Conclusions

Functional nanocomposites have transitioned from simple physical mixtures into highly tailored, multi-dimensional material systems engineered at the atomic scale. By combining complementary phases, these architectures generate synergistic properties that overcome the thermodynamic constraints of single-component systems. This structural control enables order-of-magnitude improvements in solar harvesting, carrier separation, electrochemical charge storage, and selective contaminant extraction. To bridge the gap between academic research and commercial deployment, future research must move beyond iterative material synthesis. Instead, the field should leverage high-throughput computational modeling, machine-learning-driven structure predictions, and operando analytical tools to systematically optimize interfacial properties. Overcoming the challenges of large-scale manufacturing and long-term environmental safety will establish functional nanocomposites as core technologies for global sustainable energy and environmental infrastructure.

Acknowledgment

The authors express their sincere gratitude to the Principal and the Department of Chemistry, Guruji Vidnyan Mahavidyalaya, Mangalwedha, for providing the necessary academic environment and institutional support for the preparation of this review article. The authors also acknowledge the valuable contributions of researchers and scientists whose published works have served as the foundation for this comprehensive review.

Financial support and sponsorship

Nil.

Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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