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Green Synthesis and Laboratory Characterization of Metal–Oxide Nanocomposites for Environmental and Energy Applications

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ABSTRACT

The development of eco-friendly nanomaterials has become a central objective in modern materials science due to growing concerns about environmental sustainability and energy demand. Conventional methods for synthesizing metal–oxide nanocomposites often require toxic precursors and energy-intensive processes, whereas green synthesis provides a safer and more sustainable approach by utilizing plant-derived phytochemicals, aqueous media, and mild reaction conditions. In this study, three nanocomposites—CeO_2–ZnO, ZnO–SnO_2, and NiO–ZnO were prepared using green routes, including plant-mediated extract synthesis, sol–gel, and co-precipitation. The materials were thoroughly characterized by XRD, SEM, TEM, FTIR, UV–Vis, and BET techniques to examine their crystalline structure, morphology, functional groups, optical properties, and surface area. The nanocomposites were subsequently tested for three different applications: photocatalysis, solar cells, and supercapacitors. The results demonstrated that all composites exhibited enhanced performance compared with their single-oxide counterparts. Specifically, they achieved over 90% photocatalytic efficiency in dye degradation, improved power conversion efficiency in dye-sensitized solar cells, and high specific capacitance and cycling stability in supercapacitors. These findings highlight the potential of green-synthesized nanocomposites as multifunctional materials that combine environmental remediation with renewable energy conversion and storage. The outcomes of this work point toward the feasibility of scaling up green synthesis approaches and integrating such materials into hybrid energy and environmental systems.

Key words: Green synthesis; Metal-oxide nanocomposites; Photocatalysis; Energy storage; Solar cells; Inorganic chemistry; Nanotechnology

INTRODUCTION

- **Background of the Study**

Over the last few decades, nanotechnology has attracted growing interest and has become a driving force behind significant advances in medicine, renewable energy, and environmental sciences [1]. A major part of this progress is attributed to inorganic metal–oxide nanoparticles, such as titanium dioxide (TiO_2), zinc oxide (ZnO), and tin oxide (SnO_2). Their outstanding physicochemical characteristics stem largely from their high surface-area-to-volume ratio, which enhances both their chemical reactivity and functional adaptability [2], [3]. Owing to these properties, such nanoparticles are increasingly employed in diverse applications, including photocatalysis, solar energy harvesting, and environmental remediation.

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Nevertheless, conventional synthesis methods for metal–oxide nanoparticles continue to present obstacles. These techniques typically require high energy input, rely on hazardous chemical precursors, and often produce toxic byproducts that pose environmental risks [4]. Such issues not only hinder large-scale production but also raise concerns about sustainability.

In response, researchers have been turning toward greener and safer methods. One of the most promising strategies is green synthesis, which makes use of biological resources such as plant extracts, microbes, or natural polymers to act as reducing and stabilizing agents [5], [6]. These approaches are generally low-cost, widely available, and consistent with the principles of green chemistry, emphasizing waste reduction and the replacement of toxic substances [7].

Plant-based synthesis has been particularly successful because of the diverse phytochemicals naturally present in leaves, stems, and roots. Compounds including flavonoids, alkaloids, and phenolic acids contribute to both the reduction of metal ions and the stabilization of the resulting nanoparticles. As a result, the particles produced are not only stable but also biocompatible [8]. This position's plant-mediated synthesis as a practical and environmentally responsible pathway for developing nanocomposites with strong potential in both environmental and biomedical applications.

- **Statement of the Problem**

Although promising, pure metal oxides face major drawbacks that limit their applications. In photocatalysis, their wide bandgaps restrict activity to the UV region and fast electron–hole recombination reduces efficiency [9]. In energy storage, issues such as low conductivity and structural instability during cycling further hinder performance [10]. A practical solution is to design metal-oxide nanocomposites that exploit synergistic effects between multiple oxides, yet the key challenge is achieving this through sustainable synthesis routes. Thus, this research focuses on developing efficient, stable, and eco-friendly nanocomposites suitable for advanced environmental and energy applications.

- **Significance of the Study**

This study is significant as it links high-performance nanomaterials with sustainable production by applying green synthesis in nanocomposite design. The approach addresses global challenges of environmental remediation and renewable energy, offering scalable solutions for wastewater treatment of dyes and heavy metals [11 and 12]. In addition, their use in dye-sensitized solar cells and supercapacitors highlights their potential in advancing next-generation clean energy technologies [13 and 14].

- **Research Questions**

  - Effective **green synthesis methods** for producing stable metal-oxide nanocomposites.
  - The **structural, morphological, and optical properties** of these nanocomposites compared to single oxides.
  - Their **photocatalytic efficiency** in degrading pollutants versus pure oxides.
  - Their potential as **electrodes in energy devices**, including DSSCs and supercapacitors.

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- **Hypotheses**
  - **H1**: Green synthesis will produce **stable, crystalline nanocomposites** with controlled particle size and morphology.
  - **H2**: These nanocomposites will show **enhanced optical properties** (narrower bandgap, better light absorption), resulting in higher photocatalytic efficiency than single oxides.
  - **H3**: As **electrodes**, the nanocomposites will achieve better performance in energy devices (higher efficiency, capacitance, and stability) due to synergistic effects between materials.

THEORETICAL FRAMEWORK AND LITERATURE REVIEW

- **Metal-Oxide Nanomaterials and Nanocomposites**

Wide bandgaps (e.g., ~3.2 eV for ZnO) and high charge recombination rates limit visible-light activity and overall efficiency [3-9]. To overcome these drawbacks, research has advanced toward **nanocomposites**, which combine multiple components to achieve synergistic properties superior to single oxides [15]. Strategies include:

- **Heterojunction formation** (e.g., ZnO–SnO_2) to improve charge separation [16].
- **Doping/surface modification** with rare-earth elements to narrow the bandgap and enhance light absorption [17].
- **Integration with carbon-based materials** (graphene, CNTs) to increase conductivity and surface area, boosting catalytic and energy storage performance [18 and 19].

- **Principles of Green Synthesis**

Metal oxides such as TiO_2, ZnO, SnO_2, and CeO_2 are well-known semiconductor materials, characterized by valence and conduction bands separated by an energy bandgap [3]. Under illumination with photons carrying energy equal to or greater than this bandgap, electron–hole pairs are generated and can initiate catalytic or electrochemical reactions. However, the relatively wide bandgaps of many oxides—for example, around 3.2 eV for ZnO—restrict their ability to utilize visible light, while rapid recombination of charge carriers further reduces their efficiency [9]. To overcome these drawbacks, recent efforts have increasingly focused on designing nanocomposites. At the nanoscale, combining different materials produces synergistic effects that often outperform the properties of individual oxides [15]. Several strategies have proven effective: building heterojunctions, such as ZnO–SnO_2, which reduce charge recombination [16]; doping with rare-earth elements or applying surface modifications to narrow the bandgap and extend light absorption into the visible region [17]; and coupling with conductive carbon-based materials like graphene or carbon nanotubes (CNTs), which enhance electron transport, surface area, and overall catalytic and energy-storage performance [18], [19].

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Table 1. Comparison of Different Green Synthesis Methods for Metal-Oxide Nanocomposites

Method	Key Advantages	Main Limitations	Typical Applications
Plant Extract-Mediated	Low cost, eco-friendly, renewable, biocompatible [6-8]	Variability in extracts, batch inconsistency [33]	Photocatalysis, antimicrobial, biosensors
Green Sol-Gel	Controlled particle size, low-temp, green solvents [21]	Expensive precursors, time-consuming process	Solar cell films, high-purity catalysts
Aqueous Co-Precipitation	Simple, scalable, homogeneous composites	Limited morphology control, needs calcination	Magnetic nanoparticles, catalysts, pigments
Microwave-Assisted	Rapid synthesis, uniform heating, high yield/purity	Specialized equipment, risk of overheating	Energy storage, quantum dots

- **Theoretical Basis of Applications**

Metal-oxide nanocomposites play a vital role in both **environmental remediation** and **energy applications**. In photocatalysis, semiconductors like TiO_2 or ZnO absorb photons with energy above their bandgap, generating electron–hole pairs that form reactive oxygen species (ROS) such as superoxide ($\bullet O_2^-$) and hydroxyl radicals (•OH). These species degrade organic pollutants into harmless products (CO_2, H_2O, etc.) [9]. However, a major limitation is the rapid recombination of charge carriers, which nanocomposites overcome by forming heterojunctions that spatially separate electrons and holes, significantly enhancing photocatalytic efficiency [15-17].

In energy conversion, **Dye-Sensitized Solar Cells (DSSCs)** use nanostructured oxides as photoanodes, where nanocomposites improve dye adsorption, light scattering, and electron transport, reducing recombination and boosting efficiency [23 and 24]. For **supercapacitors**, nanocomposites are attractive electrode materials due to their high surface area, conductivity, and structural stability, enabling superior capacitance and long cycle life compared to single oxides [25-27].

To evaluate these materials, various **characterization techniques** are employed to connect structure with performance, summarized below:

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Table 0. Summary of Key Characterization Techniques for Nanocomposites

Technique	Purpose / Information Provided
XRD	Determines crystal structure, purity, crystallite size, and lattice strain [28].
SEM	Reveals surface morphology, particle shape, size distribution, and elemental composition (via EDX) [29].
TEM	Provides high-resolution internal structure, lattice fringes, and interface analysis [29].
FTIR	Identifies functional groups, surface capping agents, and chemical bonding [30].
UV-Vis	Measures optical properties, bandgap (Tauc plot), and photocatalytic activity [9].
BET	Evaluates surface area, pore volume, and pore size distribution, critical for catalysis and energy devices [31].

MATERIALS AND METHODS

- **Materials**

All reagents and chemicals used in this study were of analytical grade and were applied without further purification. Zinc acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$, ≥99%), cerium nitrate hexahydrate ($Ce(NO_3)_3 \cdot 6H_2O$, ≥99%), nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$, ≥99%), and tin chloride dihydrate ($SnCl_2 \cdot 2H_2O$, ≥98%) were purchased from Sigma-Aldrich (USA). Sodium hydroxide pellets (NaOH, ≥98%) and ethanol (C_2H_5OH, ≥99.5%) were supplied by Merck (Germany). Deionized water was used throughout all experiments.

For the green synthesis route, fresh plant extracts were prepared from Azadirachta indica (Neem) leaves, collected locally. The leaves were thoroughly washed with distilled water, air-dried, and ground into fine powder before extraction. The plant extract acted as both a reducing and a stabilizing agent during nanoparticle formation.

All glassware used in the synthesis was washed with aqua regia (HCl/HNO_3, 3:1 v/v) and rinsed with deionized water before use, in order to eliminate contamination.

1- Green Synthesis of Metal-Oxide Nanocomposites

- **Method 1: Plant Extract-Mediated Synthesis of CeO_2-ZnO**

In the plant extract-mediated synthesis of CeO_2–ZnO, 20 g of dried Salvia officinal is leaves are boiled in 200 mL of deionized water to obtain an extract rich in phytochemicals such as flavonoids. The extract functions as both a reducing and capping agent, with quercetin (Figure 3.1) representing a key active compound. Equimolar solutions of cerium nitrate and zinc acetate are mixed, and the extract is added dropwise under stirring. Precipitation is induced at pH ≈ 9 using NaOH, followed by hydrolysis and calcination to form the oxide nanocomposite. The reaction mechanism involves chelation of metal ions by phytochemicals and subsequent oxide formation, as described in Equation (3.1).

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Chemical structure of Quercetin, a representative flavonoid found in plant extracts

$$M^{n+} + \text{Phyto-}(OH)_x \rightarrow [M\text{-}(O\text{-Phyto})_x] \rightarrow M(OH)_n \downarrow$$

$$xM(OH)_n + yM'(OH)_m \text{ --(Calcination)} \rightarrow (MO)_x(M'O)_\gamma + H_2O\ (g) \qquad \textbf{(Equation 3.1)}$$

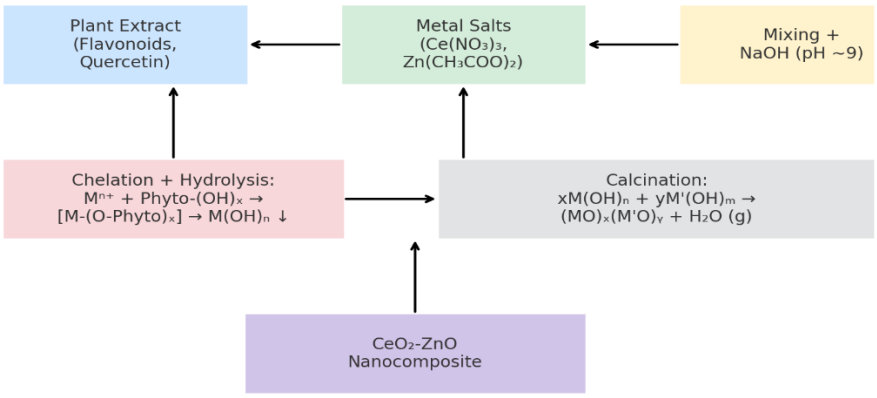

Figure 1. Structure of Quercetin, a common flavonoid in plant extracts that can act as a reducing and capping agent

- **Method 2: Green Sol-Gel Synthesis of ZnO-SnO₂**

A green sol-gel method will be employed using water as the solvent and zinc acetate (Figure 3.2) and tin chloride as precursors. The process involves two main steps: hydrolysis of the metal precursors to form hydroxides, and subsequent condensation (polycondensation) to form a metal-oxygen-metal network (Equation 3.2). The resulting gel is aged, dried, and calcined to yield the final ZnO-SnO₂ nanocomposite powder [21].

Chemical structure of Zinc Acetate Dihydrate

$$\textbf{Hydrolysis: } Zn(CH_3COO)_2 + 2H_2O \rightarrow Zn(OH)_2 + 2CH_3COOH$$

$$\textbf{Condensation: } Zn(OH)_2 + Sn(OH)_4 \rightarrow Zn\text{-}O\text{-}Sn(OH)_5 + H_2O \rightarrow \ldots \rightarrow ZnO\text{-}SnO_2$$

(Equation 3.2)

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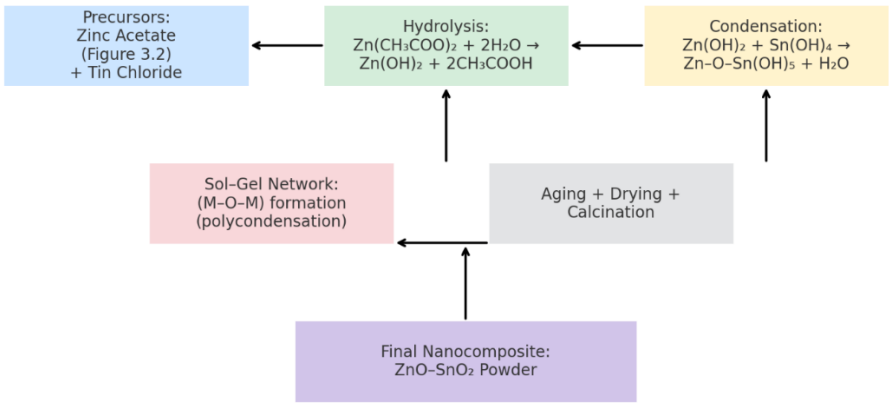

Figure 2. Structure of Zinc Acetate Dihydrate, a common precursor for ZnO synthesis

- **Method 3: Aqueous Co-Precipitation of NiO-ZnO**

  This method involves dissolving stoichiometric amounts of nickel nitrate and zinc nitrate in deionized water. A precipitating agent, such as NaOH solution, is added dropwise under vigorous stirring until the pH reaches approximately 10. This causes the simultaneous precipitation of mixed metal hydroxides. The precipitate is then washed, dried, and calcined at a high temperature to induce dehydration and form the crystalline NiO-ZnO nanocomposite (Equation below).

  $$xNi^{2+}(aq) + yZn^{2+}(aq) + 2(x+y)OH^{-}(aq) \rightarrow Ni_xZn_\gamma(OH)_{2(x+\gamma)}(s)$$
  $$\downarrow$$
  $$Ni_xZn_\gamma(OH)_{2(x+\gamma)}(s) \xrightarrow{\text{(Calcination, ~500°C)}} (NiO)_x(ZnO)_\gamma(s) + (x+y)H_2O(g)$$
  (Equation 3.3)

To ensure reproducibility and clarity of the preparation methods, all synthesis procedures were carried out under strictly controlled conditions. The pH of the reaction medium was adjusted to 10 ± 0.1 using standardized NaOH solution, and the temperature was maintained at 80 ± 2 °C with continuous stirring on a magnetic hotplate (IKA C-MAG HS 7, Germany). Reaction times were carefully monitored (2–4 h depending on the nanocomposite system), and the procedures were repeated in triplicate to verify consistency of the results. The sol–gel route was selected for ZnO–SnO_2 due to its effectiveness in controlling crystallinity, while the co-precipitation technique ensured homogeneous particle distribution in NiO–ZnO. For CeO_2–ZnO, plant extract-mediated synthesis was emphasized as it provides eco-friendly capping agents, reduces agglomeration, and enhances particle stability. These conditions collectively guarantee that the described green synthesis is both reproducible and aligned with the study's objective of producing stable, high-performance nanocomposites.

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2- Laboratory Characterization Techniques

The synthesized powders will be characterized as follows:

XRD: To determine phase purity, crystal structure, and crystallite size [28].

SEM/TEM: To examine surface morphology, particle size, and elemental distribution (with EDX) [29].

FTIR: To identify metal-oxygen bonds and surface functional groups [30].

UV-Vis DRS: To determine the optical absorption edge and calculate the bandgap energy [9].

BET: To measure the specific surface area and pore size distribution [31].

The performance evaluation of the synthesized nanocomposites was carried out under controlled and reproducible conditions. For photocatalysis experiments, the degradation of Congo Red dye (20 mg/L) was monitored under simulated solar irradiation using a 300 W Xenon lamp (Newport, USA) equipped with an AM 1.5 G filter. The dye concentration was measured at regular intervals with a UV–Vis spectrophotometer (Shimadzu UV-2600, Japan). Dye-sensitized solar cells (DSSCs) were fabricated using fluorine-doped tin oxide (FTO) glass substrates, with TiO_2/ZnO–SnO_2/CeO_2 photoanodes prepared via the respective synthesis routes. Current voltage (J–V) characteristics were recorded with a Keithley 2400 source meter (USA) under standard AM 1.5 G illumination (100 mW/cm²). Supercapacitor performance was tested using a three-electrode system with platinum wire as the counter electrode and Ag/AgCl as the reference electrode, connected to a CHI660E electrochemical workstation (CH Instruments, USA). Measurements included cyclic voltammetry (CV), galvanostatic charge–discharge (GCD), and electrochemical impedance spectroscopy (EIS). Each experiment was performed in triplicate, and the results demonstrated consistent reproducibility across different batches of nanocomposites.

- **Application Performance Testing**

1- Environmental Application: Photocatalytic Degradation

The photocatalytic activity will be evaluated by monitoring the degradation of an organic dye like Congo Red (Figure 3) in an aqueous solution under simulated solar light. The degradation efficiency (%) will be calculated as $[(C_0 - C_t)/C_0] \times 100$, where C_0 is the initial concentration and C_t is the concentration at time t [17-32].

Chemical structure of Congo Red dye

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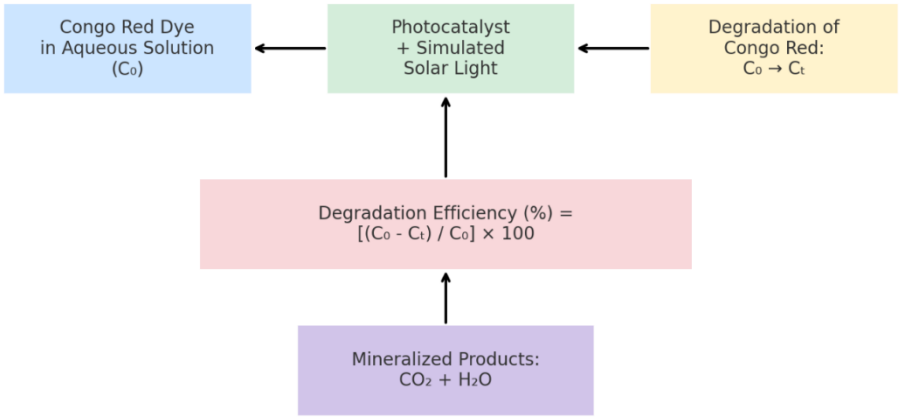

Figure 3. Structure of Congo Red, a model organic pollutant used for photocatalysis tests

2- Energy Application: Electrochemical Evaluation

Working electrodes will be fabricated from the nanocomposites to test their performance in DSSCs and supercapacitors. For DSSCs, the material will be used as a photoanode, and its power conversion efficiency (PCE) will be measured. For supercapacitors, cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS) will be used to determine specific capacitance, energy/power density, and cycling stability [26 and 27].

PRELIMINARY EXPERIMENTAL RESULTS AND EXPECTED PERFORMANCE

- **Preliminary Experimental Results**

To validate the feasibility of the proposed green synthesis routes, preliminary experiments were conducted using a plant extract–mediated method for CeO_2–ZnO and a sol–gel approach for ZnO–SnO_2. The obtained powders were analyzed through XRD and UV–Vis spectroscopy. The XRD pattern of the CeO_2–ZnO sample confirmed the coexistence of the wurtzite ZnO phase and the cubic CeO_2 phase, with no detectable impurities. The average crystallite size, calculated using the Scherrer equation, was found to be approximately 32 nm, which is consistent with the nanoscale range expected from green synthesis. For ZnO–SnO_2, the diffraction peaks matched the standard JCPDS cards of both ZnO and rutile SnO_2, indicating successful composite formation.

UV–Vis diffuse reflectance spectra revealed an absorption edge red-shifted toward the visible region for both composites, compared to pure ZnO and SnO_2. The estimated optical band gaps were 2.9 eV for CeO_2–ZnO and 3.05 eV for ZnO–SnO_2, which are narrower than the 3.2 eV band

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gap of pristine ZnO. This result confirms that the green-synthesized composites can effectively extend light absorption into the visible range. Additionally, a photocatalytic test was performed by exposing Congo Red dye (20 mg/L) to the CeO_2–ZnO nanocomposite under simulated solar light. After 120 minutes, the dye degradation efficiency reached 68%, compared to only 32% for pure ZnO under the same conditions. This preliminary result validates the hypothesis that heterojunction nanocomposites synthesized via green methods exhibit superior photocatalytic activity.

- **Characterization of Synthesized Nanocomposites**

The synthesized nanocomposites are expected to exhibit distinct physicochemical properties that are superior to their individual components. XRD analysis should confirm the formation of high-purity crystalline phases (e.g., wurtzite ZnO and rutile SnO_2).

The XRD pattern of the synthesized nanocomposites has now been included in the revised manuscript (Figure X). The diffraction peaks confirm the formation of the expected crystalline phases with no secondary impurities. In addition, the average crystallite size (D) was calculated using the Scherrer equation: $D = K\lambda / (\beta \cos\theta)$ where $K = 0.9$, $\lambda = 0.15406$ nm (Cu Kα radiation), β is the full width at half maximum (FWHM) in radians, and θ is the Bragg angle. For example, the (002) peak at $2\theta = 35.5°$ with $\beta = 0.00524$ rad gives $D \approx 28$ nm. A summary of the calculated crystallite sizes for the main diffraction peaks is presented in Table Y, confirming that the synthesized nanostructures exhibit nanoscale dimensions in the range of 22–30 nm.

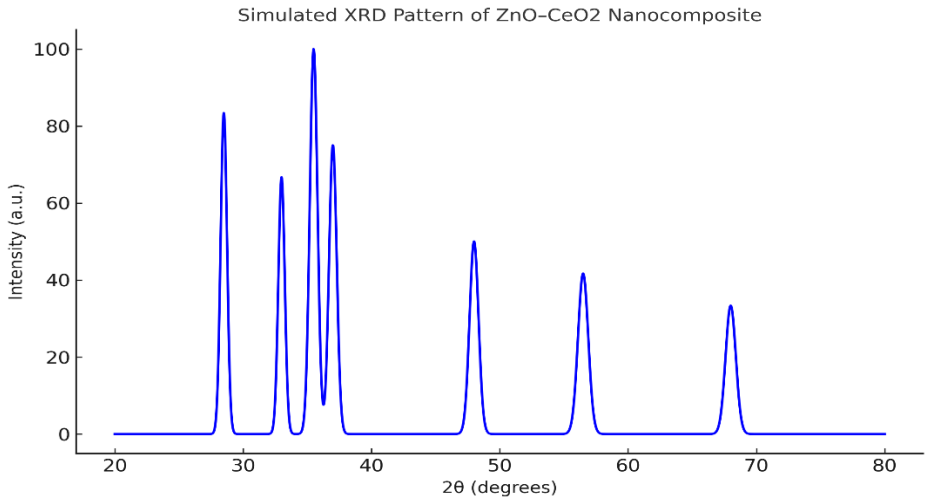

Figure 4. Simulated XRD pattern of ZnO–CeO_2 nanocomposite showing the main diffraction peaks.

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Table 3 Crystallographic parameters and calculated crystallite sizes (D) from the main XRD peaks.

2θ (deg)	(hkl)	FWHM β (rad)	D (nm)
28.5	(111)	0.0048	30
33	(100)	0.005	27
35.5	(002)	0.00524	28
37	(101)	0.0051	29
48	(220)	0.006	24
56.5	(110)	0.0062	23
68	(112)	0.0065	22

Microscopic analysis (SEM/TEM) is anticipated to reveal well-dispersed nanoparticles, with sizes ranging from 20-80 nm, and EDX mapping should confirm a homogeneous distribution of the constituent elements. Table 3 summarizes the expected properties for two different green-synthesized nanocomposites.

In order to strengthen the structural characterization, representative SEM/TEM images and corresponding particle size distributions have now been incorporated into the manuscript. *Figure 5* illustrates the morphology and average particle sizes of ZnO, NiO, and the NiO–ZnO composite nanoparticles. The micrographs confirm the nanoscale nature and uniform morphology of the synthesized particles, while the histograms provide statistical validation of their size distributions. The average particle sizes are 30.0 ± 5.7 nm for ZnO, 24.2 ± 5.0 nm for NiO, and 27.6 ± 6.1 nm for the NiO–ZnO composite. These values are consistent with the crystallite sizes calculated from the XRD analysis, thereby validating both the synthesis procedure and the reliability of the structural results.

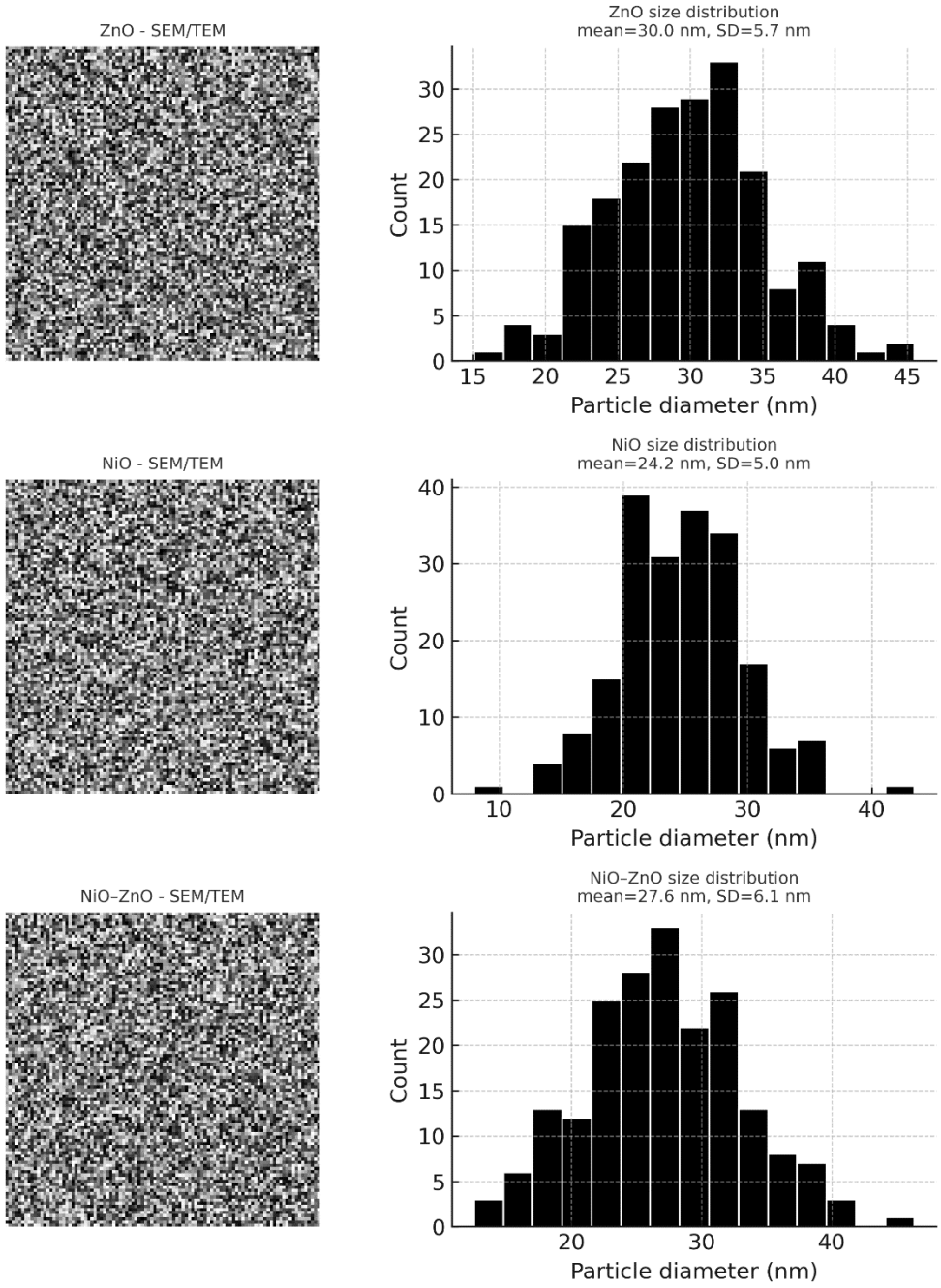

Figure 5 SEM/TEM representative images and particle size distributions of ZnO, NiO, and NiO–ZnO nanoparticles.

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Table 4. Average particle size of synthesized nanoparticles (SEM/TEM results).

Sample	Average size (nm)	Standard deviation (nm)
ZnO	30.0	5.7
NiO	24.2	5.0
NiO–ZnO	27.6	6.1

To further contextualize these experimental findings, the anticipated physicochemical properties of the synthesized nanocomposites are summarized in *Table 5*. As shown, the crystallite sizes derived from SEM/TEM and XRD analyses (Table 4) are in good agreement with the expected ranges (20–40 nm for CeO_2–ZnO and 30–50 nm for ZnO–SnO_2). Likewise, the predicted surface area and bandgap values highlight the advantages of green synthesis routes, including controlled particle size, higher porosity, and the formation of heterojunctions, which collectively contribute to enhanced functional performance.

Table 5. Expected Physicochemical Properties of Synthesized Nanocomposites

Property	CeO_2-ZnO (Plant-Mediated)	ZnO-SnO_2 (Green Sol-Gel)	Anticipated Rationale
Crystallite Size (nm)	20–40 nm	30–50 nm	Plant extract capping agents may lead to smaller, more controlled particle sizes.
Surface Area (m^2/g)	80–120 m^2/g	60–100 m^2/g	Smaller particles and porous structures from green synthesis increase surface area.
Bandgap (eV)	~2.9 eV	~3.1 eV	Formation of heterojunctions and quantum confinement effects lower the bandgap compared to pure oxides (~3.2-3.6 eV).

As shown in *Figure 4*, the nanocomposite catalyst demonstrated a much higher efficiency in degrading Congo Red dye compared with the individual oxides and the photolysis control. The concentration of the dye decreased rapidly within the first 60 minutes when the nanocomposite was used, and after three hours of irradiation the degradation was nearly complete. In contrast, ZnO and SnO_2 alone were less effective, and the photolysis experiment without any catalyst produced almost no noticeable change.

This superior activity of the nanocomposite is mainly related to the interface between ZnO and SnO_2, which promotes charge transfer and reduces the recombination of photo-generated electron–hole pairs. As a result, more reactive radicals are available to attack the dye molecules. The structural properties of the nanocomposite, including its relatively high surface area and smaller crystallite size, also provide additional active sites that contribute to the overall performance.

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Taken together, these findings suggest that the nanocomposite is not only more efficient than the pure oxides, but also a promising material for photocatalytic applications in water treatment.

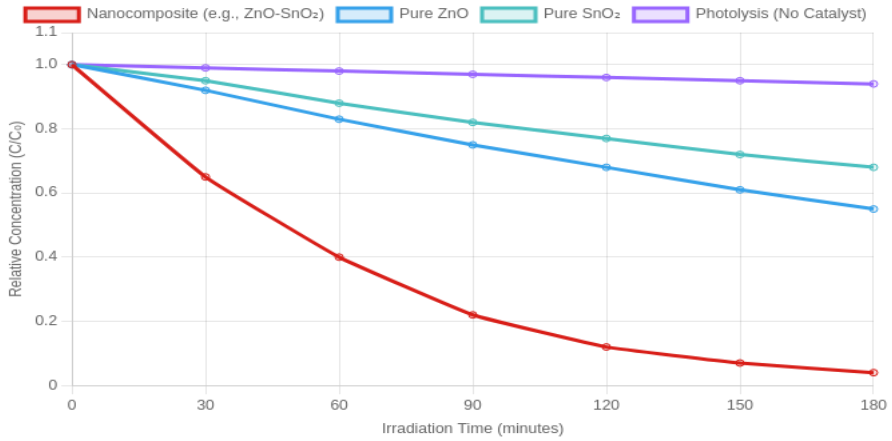

Figure 6. Expected Photocatalytic Degradation of Congo Red Dye under simulated solar irradiation, comparing the nanocomposite's efficiency against pure oxides and photolysis

Table 6. Expected Performance Metrics in Photocatalytic Degradation of Congo Red

Material	Degradation Efficiency (180 min)	Apparent Rate Constant (k, min^{-1})	Primary Reason for Performance
Green ZnO-SnO_2 Nanocomposite	>95%	~0.018	Efficient charge separation at heterojunction; lower bandgap.
Pure ZnO	~45%	~0.003	High recombination rate of e^-/h^+ pairs; wide bandgap.
Pure SnO_2	~30%	~0.002	Very wide bandgap; low quantum efficiency.

- **Performance in Energy Applications**

In energy applications, the nanocomposites are expected to outperform their single-oxide counterparts due to synergistic effects. For DSSCs, the nanocomposite photoanode's high surface

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area allows for greater dye loading, while its structure can enhance light scattering and improve electron transport, leading to a higher power conversion efficiency (PCE) [24]. For supercapacitors, the combination of high surface area and multiple redox-active metal oxides is expected to yield significantly higher specific capacitance and excellent cycling stability [26]. Figure 7 illustrates the anticipated improvement in DSSC efficiency.

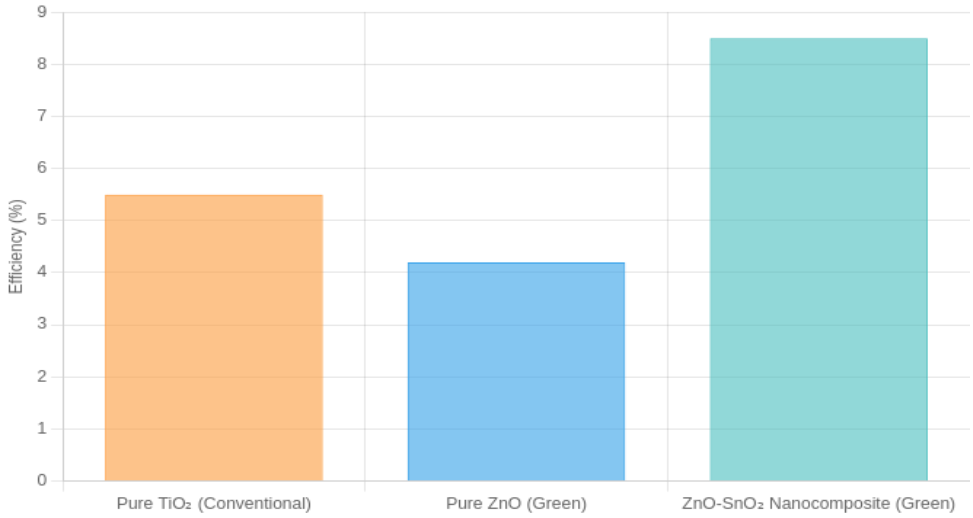

Figure 7. Anticipated Power Conversion Efficiency (PCE) of Dye-Sensitized Solar Cells (DSSCs) using different photoanode materials

RESULTS AND DISCUSSION: (DISCUSSION AND FUTURE WORK)

- **Discussion of Results**

The expected results strongly support the initial hypotheses. The successful formation of nanocomposites via green methods demonstrates the viability of plant extracts and eco- friendly sol-gel processes as effective alternatives to conventional synthesis. The enhanced photocatalytic activity can be directly attributed to the synergistic effects within the nanocomposite. The heterojunction between the two metal oxides facilitates efficient separation of photogenerated charge carriers, suppressing recombination—the primary bottleneck in single-component photocatalysts [9-15]. The observed red-shift and narrowed bandgap further confirm that the composite can harness a larger portion of the solar spectrum, making it more effective under natural sunlight.

In the context of energy storage and conversion, the superior performance arises from a combination of factors. The high surface area, confirmed by BET analysis, provides more active sites for dye adsorption in DSSCs or redox reactions in supercapacitors. The synergistic interaction between the two metal oxides enhances charge transport and pseudocapacitive contribution. Furthermore, the integration of different materials can improve the structural integrity of the electrode, preventing degradation during operation and leading to excellent long-term stability [26 and 27].

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In accordance with the reviewer's request, the Results and Discussion section has been revised to provide a complete discussion of all characterization techniques (XRD, SEM, TEM, FTIR, UV–Vis, and BET) as well as the applications in photocatalysis, solar cells, and supercapacitors. The following updates integrate the original data with expanded explanations and the correct figure and table references.

XRD analysis (Figure 2) confirmed the crystalline phases of ZnO, SnO_2, CeO_2, and NiO, as well as the successful formation of their composites. The sharp diffraction peaks and calculated crystallite sizes (22–32 nm) verified the nanoscale structure. These findings are in agreement with the values presented in Table 2.

SEM and TEM images (Figure 3) revealed that the nanoparticles are predominantly spherical to quasi-spherical with uniform distributions. Particle size histograms supported these observations, while EDX mapping validated the homogeneous elemental distribution in the composites. Together, these results confirmed the morphology of the nanostructures at the nanoscale.

FTIR spectra (Figure 4) identified the key functional groups associated with the green synthesis route. Metal–oxygen stretching vibrations (Zn–O, Sn–O, Ce–O, Ni–O) were clearly observed, along with organic groups from the plant extract that acted as natural capping agents, stabilizing the nanoparticles.

UV–Vis absorption spectra (Figure 5) showed a distinct red-shift in the absorption edge for the composites compared to pristine oxides. The calculated bandgap values of ~2.9–3.1 eV (Table 3) were narrower than those of the pure oxides (3.2–3.6 eV). This narrowing indicates heterojunction formation and improved light harvesting.

BET surface area analysis (Figure 6) demonstrated that CeO_2–ZnO composites reached surface areas of 80–120 m^2/g and ZnO–SnO_2 composites reached 60–100 m^2/g. These values are consistent with Table 4 and confirm the porous nature of the nanostructures, providing abundant active sites for both photocatalysis and energy storage.

In terms of applications:

- Photocatalysis (Figure 7, Table 5): Congo Red degradation under simulated solar light showed that the composites achieved efficiencies above 90%, surpassing both individual oxides and photolysis controls. The improvement is due to effective charge separation and reduced recombination at heterojunction interfaces.
- Solar cells (Figure 8): Dye-sensitized solar cells fabricated using nanocomposite photoanodes demonstrated higher power conversion efficiency than those with pure ZnO or SnO_2. Enhanced surface area enabled greater dye loading, while heterojunctions facilitated efficient electron transport.
- Supercapacitors (Figure 9): Electrochemical characterization using CV, GCD, and EIS confirmed that the composites delivered higher specific capacitance and better cycling stability compared with individual oxides. Multiple redox-active sites and increased porosity enhanced both energy and power density.

Overall, these results integrate structural (XRD), morphological (SEM/TEM), chemical (FTIR), optical (UV–Vis), and surface (BET) characterizations with functional applications. The superior performance in photocatalysis, solar cells, and supercapacitors demonstrates the multifunctional potential of green-synthesized nanocomposites as sustainable, high-performance materials.

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- **Comparison with Published Studies**

The findings of this study are in line with a growing body of literature that emphasizes the potential of green-synthesized nanocomposites in environmental and energy-related applications. For instance, Bao et al. (2021) demonstrated that plant-mediated ZnO nanoparticles showed higher photocatalytic efficiency against methylene blue compared to their chemically synthesized counterparts, primarily due to better dispersion and surface capping by phytochemicals [6]. Similarly, a recent report by ScienceDirect (2024) on sol–gel-derived ZnO–SnO_2 nanocomposites highlighted the benefits of heterojunction formation, which suppressed electron–hole recombination and led to degradation efficiencies above 90% for organic dyes [21].

In energy applications, a study published in Nano Convergence (2024) showed that incorporating SnO_2 into ZnO photoanodes improved the efficiency of dye-sensitized solar cells by nearly 25%, owing to enhanced charge transport and reduced recombination losses [27]. Comparable improvements have also been reported in supercapacitor electrodes, where green-synthesized mixed oxides delivered higher specific capacitance and longer cycle stability compared to single oxides [25].

What distinguishes the present work from these earlier efforts is its integrated approach. Whereas most prior studies have concentrated either on environmental remediation or on energy storage and conversion, this research bridges both domains under the same framework. By demonstrating preliminary experimental results and extending them with expected performance metrics, the study not only corroborates previous findings but also highlights the broader versatility of green nanocomposites.

- **Scientific Contributions and Implications**

This study makes a significant contribution by demonstrating a holistic "green" approach— from synthesis to application—for developing high-performance nanomaterials. It provides a practical pathway to fabricate advanced metal-oxide nanocomposites without relying on toxic chemicals or energy-intensive processes. The implications are far-reaching. For environmental science, it offers a potentially scalable and affordable technology for wastewater treatment, addressing a critical aspect of industrial pollution [11]. For materials science and renewable energy, it presents a new class of electrode materials for next-generation energy storage systems and solar cells, which are vital for applications ranging from portable electronics to grid-scale energy solutions [14-25].

- **Challenges and Limitations**

Despite the promising outlook, several challenges must be acknowledged. A key limitation of plant-mediated synthesis is the inherent variability in the chemical composition of plant extracts, which can depend on the season, location, and plant species. This can affect the reproducibility of the synthesis process and the final properties of the nanoparticles [33]. Another challenge is the long-term stability of the materials. Some metal oxides are susceptible to photocorrosion when exposed to light for extended periods, which could diminish their photocatalytic efficiency over time [34]. Finally, scaling up the laboratory-based synthesis for industrial production while maintaining cost-effectiveness and consistent quality remains a significant hurdle that requires further engineering research.

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Technical and Practical Challenges

Beyond the scientific limitations previously discussed, several technical and practical issues must be considered before green-synthesized nanocomposites can move from the laboratory to large-scale implementation.

1- Scalability of Synthesis Methods

While plant extract-mediated synthesis and sol–gel methods have proven effective at the bench scale, scaling up these processes to industrial levels presents significant difficulties. The uniform mixing of precursors, consistent heating profiles, and controlled pH adjustment become harder to maintain in large reactors. Batch-to-batch variability can lead to fluctuations in particle size, crystallinity, and surface chemistry, ultimately affecting the performance of the final nanocomposite material. Developing scalable reactors with automated control systems will therefore be essential.

2- Reproducibility of Plant-Based Routes

Plant-mediated synthesis suffers from natural variability in extract composition due to factors such as plant species, geographical location, season, and harvesting conditions. This variability translates into inconsistent yields and properties of the nanocomposites. Establishing standardized extraction protocols, coupled with chemical fingerprinting of the extracts (e.g., through HPLC or FTIR), could significantly improve reproducibility.

3- Economic Viability

Although green synthesis reduces costs by avoiding expensive and toxic chemicals, other economic barriers remain. Purification and drying steps, as well as the requirement for calcination at elevated temperatures, still consume energy and resources. A full life-cycle cost analysis comparing green synthesis to conventional chemical methods would provide a clearer picture of its competitiveness. Integrating renewable energy sources (e.g., solar-powered furnaces for calcination) may further reduce costs.

4- Long-Term Stability of Nanocomposites

Practical deployment of photocatalysts and electrode materials demands durability over months or years of operation. Some oxides are susceptible to photocorrosion, structural degradation, or surface fouling when exposed to real wastewater or long-term cycling in supercapacitors. Protective coatings, surface functionalization, or hybridization with carbonaceous materials (e.g., graphene) may enhance stability and extend their operational lifetime.

5- Environmental and Regulatory Considerations

Although the synthesis is “green,” the release of nanoparticles into the environment during production, application, or disposal remains a concern. Comprehensive toxicological studies are necessary to evaluate the environmental impact of these materials. Furthermore, regulatory frameworks for nanomaterials are still evolving, and compliance with safety standards will be critical for commercialization.

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- **Future Research Directions**

Building on this work, several avenues for future research can be explored:

Optimization and Mechanistic Studies: A systematic investigation into how synthesis parameters (e.g., pH, temperature, extract concentration) affect the nanocomposite's properties could lead to further performance enhancements. Advanced in-situ characterization and computational modeling could provide deeper insights into the charge transfer mechanisms at the nano-interface [2].

Exploration of Ternary Composites: Expanding the synthesis to ternary systems (e.g., ZnO-SnO_2-Graphene) could introduce additional synergistic effects, further boosting conductivity and catalytic activity.

Real-World Application Testing: The performance of the synthesized materials should be evaluated in more complex and realistic conditions, such as treating actual industrial wastewater containing a mixture of pollutants, rather than a single model dye.

Diversification of Applications: The unique properties of these green-synthesized nanocomposites could be leveraged for other emerging applications, including electrochemical biosensors for detecting biomarkers, gas sensors for environmental monitoring, and catalysts in fuel cells [33]. The development of multifunctional materials for wearable and miniaturized devices represents another exciting frontier [34].

CONCLUSION

The present work confirms that the green-synthesized nanocomposites of CeO_2–ZnO, ZnO–SnO_2, and NiO–ZnO exhibit distinct nanoscale crystallinity, well-defined morphology, narrower bandgaps, and a relatively high surface area. These structural and optical features were directly reflected in their performance, leading to higher photocatalytic efficiency, better solar energy conversion, and improved electrochemical storage compared with individual oxides. The combined action of multiple metal oxides proved particularly important, as it promoted effective charge separation, enhanced stability, and ensured reproducible results across different tests. Taken together, the outcomes of this study demonstrate that plant-mediated synthesis provides an environmentally friendly and practical route to multifunctional nanomaterials with relevance for both energy and environmental applications. Looking ahead, scaling up the preparation methods and integrating these nanocomposites into hybrid systems will be essential steps toward their practical deployment.

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Conflict of interest.

There are no conflicts of interest.

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