

Green synthesis of amorphous cobalt selenide using *Hedyotis diffusa* Willd. leaf extract integrated with graphitic carbon nitride for photocatalytic hydrogen peroxide production

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Abstract. Amorphous cobalt selenide (CoSe) was synthesized using *Hedyotis diffusa* Willd. leaf extract via a sol-gel method and integrated with graphitic carbon nitride (g-C₃N₄) through ultrasonication to form CoSe/g-C₃N₄ composites for photocatalytic hydrogen peroxide (H₂O₂) production. The composite was characterized using XRD, FTIR, UV-Vis, and SEM-EDS, revealing a reduced band gap of 2.75 eV compared to 2.89 eV for g-C₃N₄ and 3.01 eV for CoSe. The 10CoSe/g-C₃N₄ composite achieved an H₂O₂ concentration of 268.6 μ M under visible light irradiation, 1.44 and 10.55 times higher than pristine g-C₃N₄ and CoSe, respectively. Scavenger experiments confirmed electrons and superoxide radicals as key species in H₂O₂ generation. The composite maintained consistent photocatalytic activity over four cycles, demonstrating its potential for sustainable H₂O₂ production.

Keywords: Phyto-synthesis, *Hedyotis diffusa* Willd., g-C₃N₄, CoSe NPs, photocatalysis.

1. INTRODUCTION

The depletion of fossil fuels and environmental concerns have increased demand for sustainable energy technologies. Among hydrogen-based carriers, hydrogen peroxide (H₂O₂) is especially attractive as both an energy carrier and a green oxidant for wastewater treatment, disinfection, and chemical synthesis [1], [2]. Current H₂O₂ production relies on the anthraquinone process, which is energy-intensive and environmentally unfriendly, underscoring the need for green alternatives. Photocatalytic H₂O₂ generation using semiconductors offers a

sustainable route by directly harvesting solar energy. Effective photocatalysts require suitable band structures, visible-light absorption, efficient charge separation, and stability. Conventional semiconductors like TiO_2 and ZnO are limited by UV-only activity, whereas graphitic carbon nitride ($\text{g-C}_3\text{N}_4$) is a promising metal-free semiconductor with a 2.7 eV band gap, easy synthesis, and selective two-electron pathways for H_2O_2 production [3]. However, pristine $\text{g-C}_3\text{N}_4$ suffers from charge recombination, poor visible-light response, and few active sites.

Transition metal chalcogenides (TMCs) such as CdS , Bi_2S_3 , and SnSe possess strong light absorption, high conductivity, and catalytic activity [4], [5]. Cobalt selenide (CoSe), though less studied, provides abundant defects and active sites in its amorphous form, which can enhance charge separation and surface reactions [6]. Coupling CoSe with $\text{g-C}_3\text{N}_4$ can therefore improve light harvesting and suppress recombination. Yet, conventional CoSe synthesis often involves harsh or costly methods. Green synthesis using plant extracts offers a sustainable alternative. *Hedyotis diffusa* Willd., rich in polyphenols and flavonoids, is a promising reducing agent [7], though its use for $\text{CoSe@g-C}_3\text{N}_4$ synthesis has not been reported.

In this work, amorphous CoSe was synthesized via *Hedyotis diffusa* Willd. extract and integrated with $\text{g-C}_3\text{N}_4$ to form $\text{CoSe@g-C}_3\text{N}_4$ composites. The method is simple, cost-effective, and eco-friendly. Structural, optical, and electrochemical properties were characterized by XRD, SEM-EDS, FTIR, UV-Vis, and impedance analyses. Photocatalytic tests demonstrated superior visible-light-driven H_2O_2 production compared with pristine $\text{g-C}_3\text{N}_4$ and CoSe , attributed to enhanced light absorption, charge separation, and active sites. Mechanistic insights and recyclability confirmed the potential of $\text{CoSe@g-C}_3\text{N}_4$ for practical photocatalytic applications.

2. MATERIALS AND METHODS

2.1. Materials and chemicals

Hedyotis diffusa Willd. (commonly known in Vietnam as “cỏ lười rần” or “bạch hoa xà”) was obtained from a local herbal store. All other chemicals were purchased from Sigma-Aldrich and Xilong. Double-distilled water (DW) was used in all experiments.

Fresh *Hedyotis diffusa* Willd. leaves were collected, rinsed thoroughly with distilled water to remove surface impurities, air-dried, and then ground into a fine powder. A specific amount of the powder was dispersed in 100 mL of an ethanol–water mixture (1:1, v/v) and ultrasonicated for 1 hour. The resulting suspension was vacuum-filtered to remove insoluble residues, and the clear filtrate was stored at 4 °C for subsequent use.

2.2. Synthesis of $\text{CoSe/g-C}_3\text{N}_4$

Graphitic carbon nitride was synthesized by thermal polymerization according to a previously reported method [8]. A mixture of melamine, urea, and ammonium chloride with a mass ratio of 3:3:1 was calcined at 550 °C for 2 hours. After naturally cooling to room temperature, the obtained solid was finely ground to yield a light-yellow $\text{g-C}_3\text{N}_4$ powder.

CoSe was synthesized by a sol–gel method using *Hedyotis diffusa* Willd. leaves extract obtained via ultrasound-assisted extraction, as previously reported [8], [9]. Briefly, 30 mL of an aqueous solution of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and Na_2SeO_3 was prepared, stirred, and heated at 70°C. Subsequently, 20 mL of the plant extract was added dropwise under continuous stirring and maintained at 70°C for 2 h. The product was collected by centrifugation, wash, dried, and then calcined at 350°C for 2 h in a foil-wrapped crucible, yielding CoSe after natural cooling.

The $\text{CoSe/g-C}_3\text{N}_4$ composites were prepared by mixing 100 mg of $\text{g-C}_3\text{N}_4$ with 5 mg of CoSe using a ball mill. The mixture was then subjected to ultrasonic treatment for 1 h to obtain the composite labeled 5 $\text{CoSe/g-C}_3\text{N}_4$. The same procedure was repeated with CoSe masses of 10 and 15 mg, yielding the composites 10 $\text{CoSe/g-C}_3\text{N}_4$ and 15 $\text{CoSe/g-C}_3\text{N}_4$, respectively.

2.3. Characterization of materials

FTIR spectra were recorded using the KBr pellet method (Alpha-E, Bruker, Germany) in the range of 600–4000 cm^{-1} to identify surface functional groups. XRD patterns were obtained with $\text{CuK}\alpha$ radiation ($\lambda = 0.15406$ nm) over $2\theta = 5\text{--}80^\circ$ (D2 Phaser, Bruker, Germany) to analyze crystalline structures. UV–Vis absorption was measured from 200–800 nm with an Evolution™ 220 spectrophotometer (Thermo Scientific, USA). Sample morphology was examined by SEM/FE-SEM, and elemental composition by EDS (JSM-IT200, JEOL, Japan). Photocatalytic degradation was performed under a 50 W LED lamp (365–420 nm).

2.4. Photoproduction of hydrogen peroxide

The photocatalytic H_2O_2 generation under 50 W LED light was evaluated by suspending the catalysts in water. H_2O_2 concentration was determined by the iodometric method. At 15 min intervals, 3 mL aliquots (1 mL reaction solution, 1 mL KHP, and 1 mL KI) were collected and analyzed by UV–Vis spectroscopy. To elucidate the reaction pathway, scavenger tests were carried out using ascorbic acid (AA), isopropanol (IPA), EDTA, and mannitol.

3. RESULTS AND DISCUSSION

3.1. Characteristics of materials

Figure 1a shows the XRD patterns of the samples. $\text{CoSe@g-C}_3\text{N}_4$ composites display peaks at ~ 13 and 27.8° , indexed to the (100) and (002) planes of $\text{g-C}_3\text{N}_4$, while pure CoSe shows no distinct peaks, confirming its amorphous nature [6], [10]. Increasing CoSe loading reduces peak intensity and shifts positions to higher angles, indicating decreased interlayer spacing and successful composite formation. FTIR spectra (Figure 1b) further confirm the structural features. Pristine $\text{g-C}_3\text{N}_4$ exhibits vibrations at 810 cm^{-1} corresponding to tri-s-triazine units, in the range of $1200\text{--}1600\text{ cm}^{-1}$ assigned to C–N/C=N stretching, and between $3000\text{--}3500\text{ cm}^{-1}$ attributed to O–H/N–H groups. Pure CoSe shows weak absorption with a characteristic Co–Se band at $600\text{--}900\text{ cm}^{-1}$. The $10\text{CoSe/g-C}_3\text{N}_4$ composite retains the typical $\text{g-C}_3\text{N}_4$ vibrations with reduced intensity and slight shifts, suggesting Co–N/Se–N interactions while preserving the framework [11], [12].

Optical properties (Figure 1c) show $\text{g-C}_3\text{N}_4$ absorbs strongly in the UV region with an edge near 450 nm and a bandgap of 2.89 eV, limiting visible-light activity. CoSe has a ~ 3.01 eV bandgap, whereas $10\text{CoSe/g-C}_3\text{N}_4$ exhibits a red-shifted edge with a narrower bandgap of 2.75 eV. The enhanced visible-light absorption and interfacial charge transfer between CoSe and $\text{g-C}_3\text{N}_4$ improve charge separation, thereby boosting photocatalytic performance. Moreover, numerous studies have reported that the band gap energy of heterojunction photocatalysts is lower than that of pristine $\text{g-C}_3\text{N}_4$, such as $\text{TiO}_2\text{-g-C}_3\text{N}_4$ [13], $\text{ZnO-g-C}_3\text{N}_4$ [14], Cu-doped $\text{g-C}_3\text{N}_4$ [15], and Zn-MOF- $\text{g-C}_3\text{N}_4$ [16], indicating that this finding is consistent with previous research. In addition to the generation of photogenerated electrons, the ability to transport these electrons plays a crucial role in determining photocatalytic activity. Figure 1d shows the EIS results of the materials. CoSe exhibits the smallest semicircle, indicating excellent charge transfer properties and efficient electron mobility [17]. In contrast, $\text{g-C}_3\text{N}_4$ shows a larger semicircle, reflecting higher charge transfer resistance and slower electron transport.

Table 1. Mass compositions of the elements in a sample of CoSe and $10\text{CoSe/g-C}_3\text{N}_4$

Element	Mass compositions (%)	
	CoSe	$10\text{CoSe/g-C}_3\text{N}_4$
C	4.06 ± 0.05	32.22 ± 0.11
N	0.32 ± 0.03	52.26 ± 0.27
O	16.42 ± 0.10	3.75 ± 0.11
Co	35.64 ± 1.01	6.47 ± 0.66
Se	43.56 ± 0.31	5.30 ± 0.15

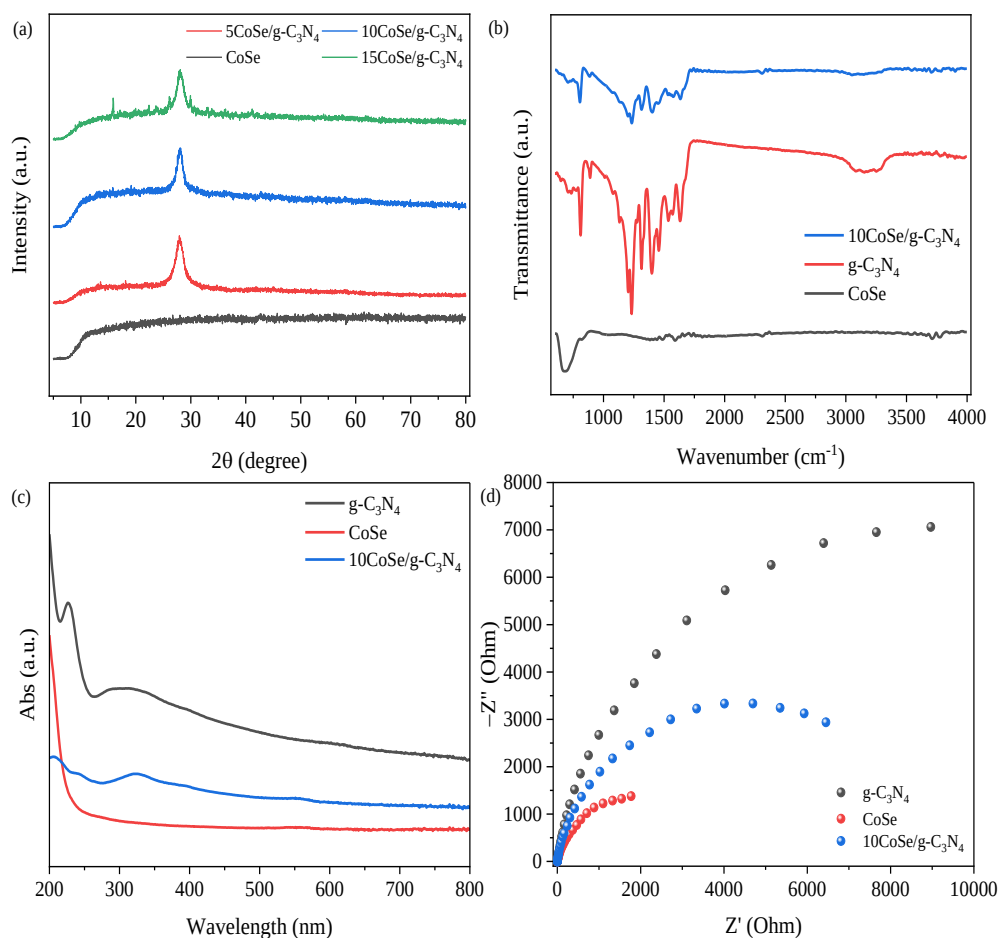


Figure 1. (a) XRD patterns, (b) FTIR spectra, (c) UV-Vis spectra, and (d) EIS spectra of material

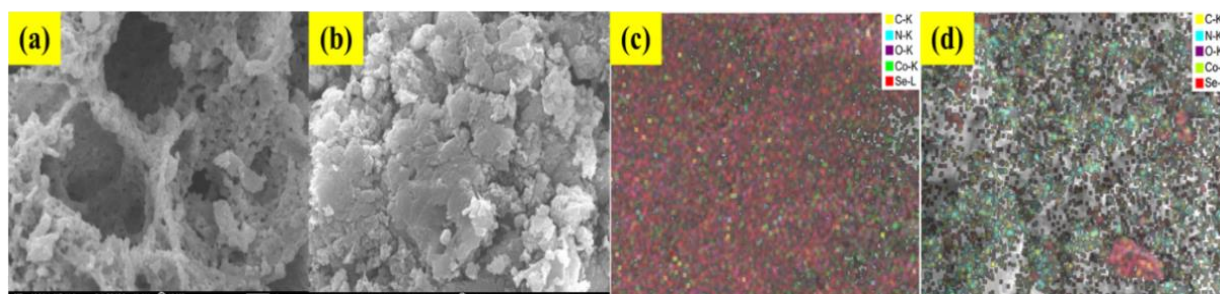


Figure 2. SEM images of (a) CoSe and (b) 10CoSe/g-C₃N₄, and elemental mapping of (c) CoSe and (d) 10CoSe/g-C₃N₄

The 10CoSe/g-C₃N₄ composite displays an intermediate arc between CoSe and g-C₃N₄, suggesting low charge transfer resistance and rapid electron transport. These results are consistent with the enhanced capability of the composite to separate and efficiently transport photogenerated charge carriers, which contributes to its superior photocatalytic performance.

SEM images (Figure 2a) reveal CoSe as irregular porous particles with honeycomb-like morphology, while plant extract assists uniform growth and prevents agglomeration. In 10CoSe/g-C₃N₄, CoSe nanoparticles are evenly dispersed on g-C₃N₄. Figure 2D shows the elemental mapping, which confirms the distributions of Co, Se, C, N, and O. Co appears as localized spots, Se is homogeneously dispersed, and C/N dominate the matrix, forming catalytic hotspots favorable for photocatalysis. Table 1 presents the mass compositions of CoSe and 10CoSe/g-C₃N₄.

3.2. Photoproduction of H₂O₂, mechanism, and reusability of the catalyst

Figure 3a shows the time-dependent H₂O₂ generation by CoSe, g-C₃N₄, and their composites. CoSe exhibits negligible activity, while g-C₃N₄ produces 111.8 μM H₂O₂, indicating moderate photocatalytic efficiency. Among the composites, 10CoSe/g-C₃N₄ performs the highest H₂O₂ concentration, whereas 5CoSe/g-C₃N₄ and 15CoSe/g-C₃N₄ show lower activities, likely due to insufficient loading or aggregation at higher contents.

Figure 3b illustrates the effect of catalyst loading on 10CoSe/g-C₃N₄. The optimum performance is obtained with 10 mg of catalyst (268.6 μM), while 5 mg limits active sites (160.9 μM) and 15 mg reduces activity (237.9 μM) due to turbidity and aggregation with ethanol as sacrificial agent. Figure 3c presents the influence of sacrificial agents. Ethylene glycol yields the highest H₂O₂ (229 μM), ethanol shows moderate activity (160.9 μM), and isopropanol exhibits poor efficiency (27.3 μM), highlighting the role of sacrificial agent selection.

Radical scavenging experiments (Figure 3d) reveal that ·O₂⁻ is the dominant species, as ascorbic acid strongly suppresses H₂O₂ production. Mannitol causes a moderate decrease, confirming ·OH involvement. EDTA enhances H₂O₂ generation by trapping holes and prolonging electron lifetime, while K₂Cr₂O₇ suppresses H₂O₂ formation, indicating the key role of electron transfer in O₂ reduction. Overall, electrons drive the formation of ·O₂⁻, the primary intermediate responsible for H₂O₂ photogeneration.

The proposed mechanism was further analyzed by estimating the band edge potentials of CoSe and g-C₃N₄ using the Mulliken electronegativity method as show in Equations (1) and (2) [18].

$$E_{CB} = \chi - E_c - 0.5E_g \quad (1)$$

$$E_{VB} = E_{CB} + E_g \quad (2)$$

where χ is the absolute electronegativity of the semiconductor; I is the ionization energy; A is the electron affinity; E_{CB} and E_{VB} are the edge potentials of conduction and valence band, respectively; E_c is the energy of free electrons on the hydrogen scale (4.5eV); and E_g is the measured band gap.

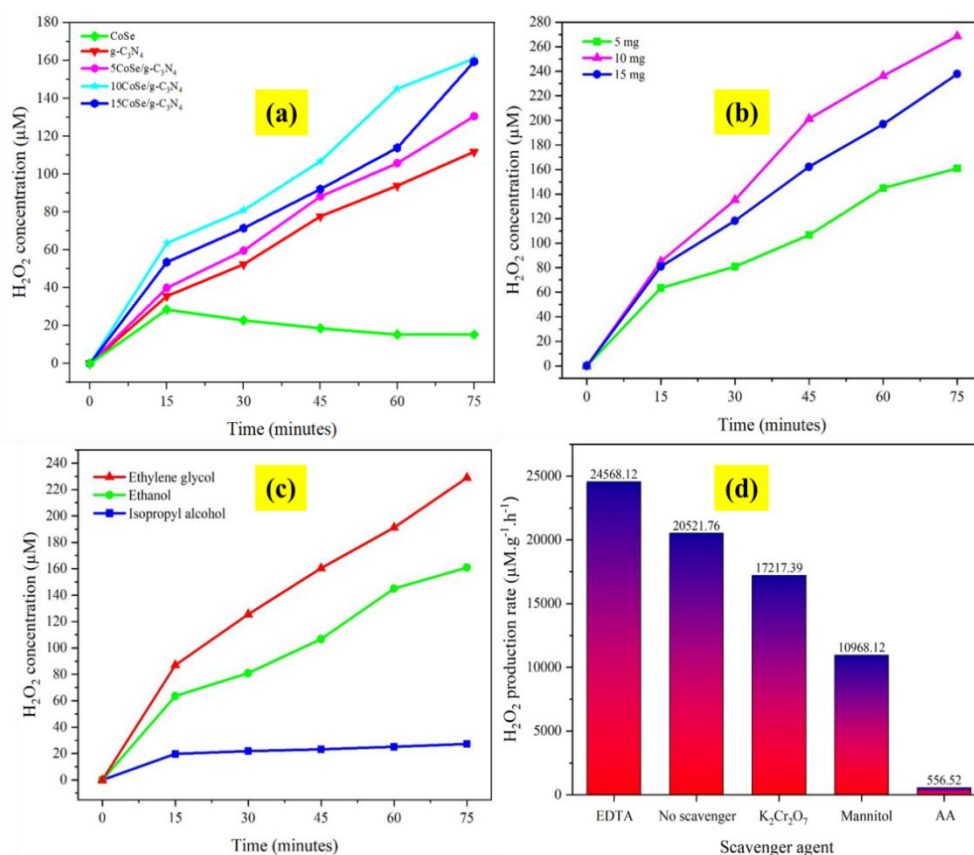


Figure 3. Effects on photocatalytic H₂O₂ generation: (a) CoSe loading, (b) catalyst dosage,

(c) sacrificial agents, and (d) radical scavengers.

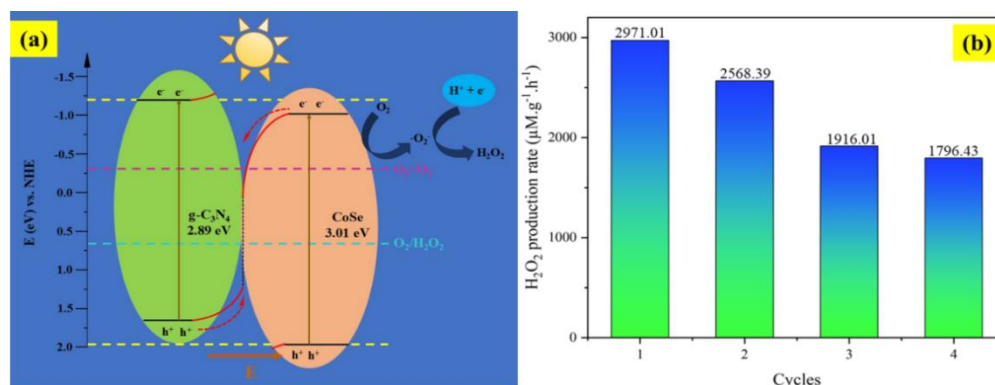


Figure 4. (a) Proposed photocatalytic mechanism of the S-scheme CoSe/g-C₃N₄ heterojunction and (b) recyclability of the composite catalyst for H₂O₂ production.

The average electronegativity (χ) of CoSe was calculated as 4.98 eV, while that of g-C₃N₄ is 4.73 eV. The conduction band (E_{CB}) of g-C₃N₄ (-1.22 eV) is more negative than that of CoSe (-1.03 eV) and the O₂/·O₂⁻ redox potential ($E^0 = -0.33$ eV vs. NHE), enabling electron transfer for O₂ reduction to ·O₂⁻. Meanwhile, the valence band (E_{VB}) of CoSe is more positive than that of g-C₃N₄, providing holes with stronger oxidation ability to react with sacrificial agents such as EDTA. This suppresses charge recombination, extends electron lifetime in g-C₃N₄, and enhances ·O₂⁻ generation and overall H₂O₂ production through the formation of an S-scheme heterojunction.

Based on radical scavenging results and band alignment, an S-scheme mechanism is proposed (Figure 4a). Under light irradiation, electrons in CoSe's conduction band recombine with holes in g-C₃N₄ valence band, while the remaining electrons in g-C₃N₄ reduce O₂ to ·O₂⁻. Holes in CoSe interact with EDTA, further suppressing recombination and enabling efficient H₂O₂ generation of up to 24.5 mM·g⁻¹·h⁻¹. This mechanism accounts for the improved photocatalytic performance by preserving strong redox potentials of the charge carriers.

The reusability of 10CoSe/g-C₃N₄ was evaluated over four cycles (Figure 4b). H₂O₂ production decreased from 2971.01 to 1796.43 μM·g⁻¹·h⁻¹, corresponding to 60.5% retention after the fourth cycle. The decline may result from catalyst loss, surface fouling, or deactivation, yet the composite maintains more than half of its initial activity, highlighting its promising stability for practical applications.

4. CONCLUSIONS

This study demonstrates the green synthesis of CoSe/g-C₃N₄ composites using *Hedyotis diffusa* Willd. leaf extract, achieving a reduced band gap (2.75 eV), enhanced visible-light absorption, and efficient charge separation. The optimized 10CoSe/g-C₃N₄ composite exhibited superior photocatalytic H₂O₂ production (268.6 μM under visible light), driven by an S-scheme heterojunction and superoxide radicals. Despite a decline to 60.5% activity after four cycles, the composite shows promising stability. These findings highlight the potential of green-synthesized CoSe/g-C₃N₄ for sustainable H₂O₂ production, with applications in energy storage, environmental remediation, and chemical synthesis. Future work should focus on improving long-term stability and scaling up the synthesis process.

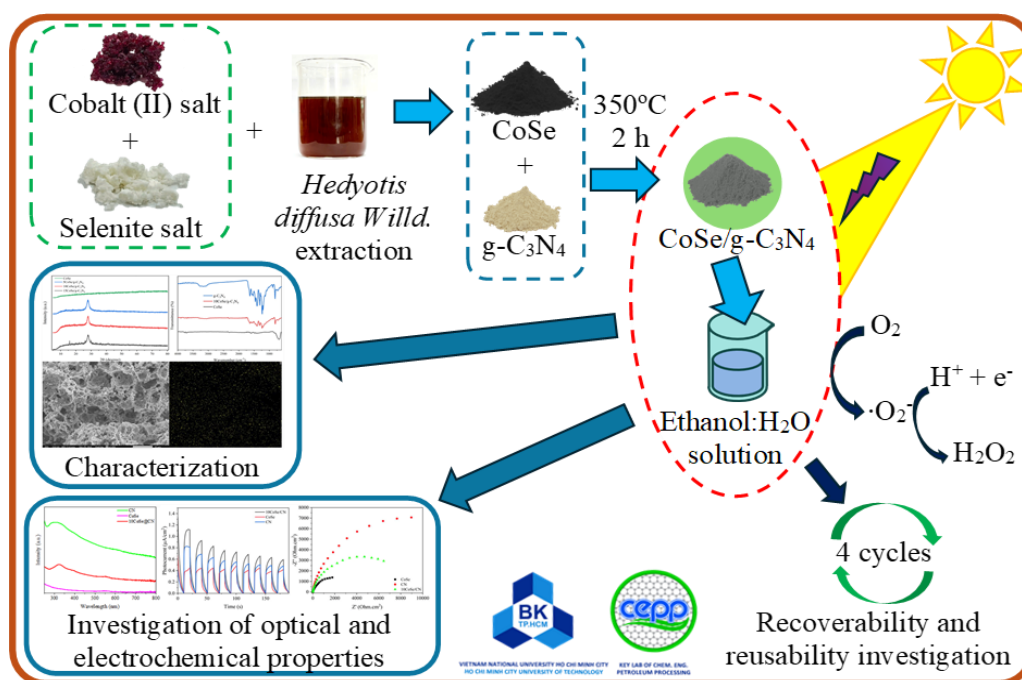
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Graphical abstract