

Article

Engineered NiCo₂O₄ Spinel Nanostructures for Enhanced Electrocatalytic Performance in Energy Storage and Non-Enzymatic Glucose Detection

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Abstract

The development of multifunctional nanostructured catalysts with high electrochemical activity and stability is crucial for sustainable technologies. Herein, we report the synthesis of CTAB-capped NiCo₂O₄ (CNC) spinel nanostructures via a facile co-precipitation method, engineered to enhance surface activity and charge transport. The optical and structural properties of the nanocomposite were confirmed by UV-Vis and TEM analysis, and the functional group present in the composite was confirmed by FT-IR study. The cubic spinel phase of the CNC was confirmed by XRD analysis. The band gap value was determined to be 2.15 eV, which confirmed the semiconductor nature of the nanocomposite. The photocatalytic degradation efficiency was achieved up to approximately 97% against malachite green. Additionally, CNC demonstrated excellent electrocatalytic performance in non-enzymatic glucose detection, exhibiting high sensitivity and reproducibility across a broad concentration range. Hence, the CNC acted as a potent oxidant for photoelectrochemical reactions.

Keywords: morphology; glucose detection; malachite green; oxidant; photo-electrochemical reaction



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1. Introduction

The pollution of water sources with various contaminants has reached concerning proportions due to the worldwide population increase and increasing industrial operations [1]. This pervasive contamination presents a substantial risk to water resources by intensifying water scarcity and compromising water quality. Approximately 15% of the colors discharged into aquatic environments via textile effluents during the dyeing process contribute to this contamination [2]. Malachite green, a durable organic dye extensively utilized in several sectors, presents significant environmental and health risks owing to its toxicity and resilience against standard degradation methods [3,4].

Glucose sensing is crucial for numerous healthcare applications, particularly for the early identification and monitoring of chronic diseases, with diabetes being the primary focus because of its status as a leading cause of death and disability globally. In 2019, the anticipated number of patients was 463 million, with forecasts indicating 10.2% growth by 2030 [5]. The first glucose biosensor was conceived in 1962 through the innovative efforts of Clark and Lyons, wherein a thin layer of concentrated glucose oxidase (GO_x) was encapsulated between two dialysis membranes, interfacing with either a pH-sensitive glass or a pO_2 -detecting electrode [6,7].

Multiple methodologies encompassing physicochemical and biological processes have been devised for dye treatment, each presenting distinct advantages and disadvantages. Photocatalytic degradation is a well-established and efficient method for the full decomposition of organic molecules found in polluted wastewater. This procedure produces highly reactive hydroxyl ($\cdot\text{OH}$) radicals by utilizing a suitable photocatalyst and light radiation. These radicals can convert water contaminants into comparatively innocuous end products, including CO_2 , H_2O , and other inorganic ions. For instance, Mahmoodi et al. demonstrated dye removal using chitosan in binary systems [8], while Kumar et al. highlighted the role of green-synthesized gold nanoparticles for catalytic applications [9]. Singh and Dhaliwal reported the photocatalytic efficiency of biosynthesized silver nanoparticles [10]. Alam et al. showed that bismuth oxide nanostructures could serve both in photocatalysis and electrochemical sensing [11], and Hammad et al. investigated how the WO_3 nanostructure impacts its photoelectrochemical performance [12]. These studies collectively illustrate that nanomaterials provide distinct advantages such as high surface area, tunable morphology, and multifunctional catalytic properties, rendering them highly suitable for pollutant degradation and glucose sensing.

The advancement of multifunctional nanomaterials exhibiting enhanced catalytic and sensing capabilities has attracted considerable interest in recent years. Among these, spinel oxides like nickel cobaltite (NiCo_2O_4) have emerged as viable candidates owing to their distinctive structural, electrical, and chemical features. NiCo_2O_4 , a mixed-metal oxide, demonstrates remarkable redox activity, elevated electrical conductivity, and environmental durability, rendering it an optimal material for various applications such as photocatalysis and electrochemical sensing. Surface modification and the integration of surfactants during synthesis have been extensively investigated to improve performance. Cetyltrimethylammonium bromide (CTAB), a cationic surfactant, is essential for the regulated synthesis of nanostructured materials. The incorporation of CTAB as a structural and morphological guiding agent facilitates the creation of CNC with an increased surface area and enhanced physicochemical properties [13,14].

Katubi et al. synthesized $\text{Nd-NiCo}_2\text{O}_4/\text{MXene}$ for photocatalytic degradation of malachite green and pendimethalin [15]. NiCo_2O_4 nanostructures exhibited limits of detection of glucose in the range between 0.0475 and 0.393 μM , and the sensitivity was determined to be 143 $\mu\text{A}/\text{mM}$ using cyclic voltammetry [16]. This present work presents the CNC fabrication through a basic co-precipitation process aimed at assessing its capabilities in photocatalysis and glucose detection. We evaluated the structure, light absorption characteristics, and chemical makeup of CNC using several analytical techniques, including X-ray diffraction, UV-visible spectroscopy, transmission electron microscopy, energy-dispersive X-ray spectroscopy, and Fourier-transform infrared spectroscopy.

2. Results and Discussion

2.1. Structure, Optical, and Chemical Composition of the CNC

Figure 1a illustrates the absorption maximum of the CNC. The samples were suspended in water and subjected to sonication in a bath sonicator for optimal dispersion. The $\lambda_{\max}$ value was recorded at 576 nm in the UV-Vis spectrum of CNC, mostly due to d-d electronic transitions inside the transition metal ions in the spinel structure. In NiCo_2O_4 , nickel (Ni^{2+}) and cobalt (Co^{3+}) ions occupy designated lattice positions, resulting in crystal field splitting of respective d-orbitals. This splitting enables electronic transitions among various energy levels within the d-orbitals. Sahoo et al. synthesized the $\alpha\text{-MnO}_2\text{@NiCo}_2\text{O}_4$ core-shell nanostructure, and the spinel configuration of the CNC was validated by the absorption spectra [17]. The band gap value was found to be 2.15 eV, which was found by using the following Equation (1).

$$h\nu = 1240/\lambda_{\max} \quad (1)$$

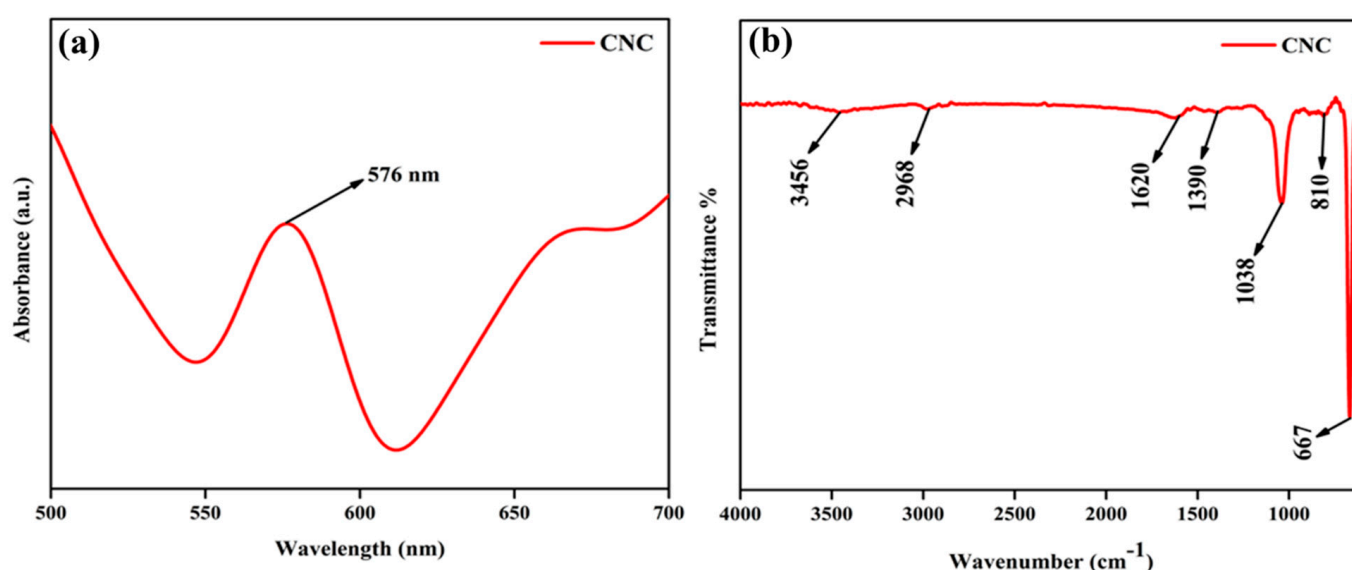


Figure 1. (a) UV-Vis spectra and (b) FTIR spectra of the CNC.

The formation of metal–oxygen bonds and the presence of functional groups in the CNC were determined through FT-IR spectroscopy (Figure 1b). The formation of the metal oxide was verified by the peak observed at 667 cm^{-1} . This peak represents the distinctive features of NiCo_2O_4 , likely attributable to the stretching vibration modes of octahedrally coordinated Co^{3+} ions and Ni^{2+} ions located at tetrahedral locations. The band observed at 1390 cm^{-1} was ascribed to the vibrational frequencies of water absorbed by the CNC or KBr. The peak at 1620 cm^{-1} was ascribed to H-O-H bending vibrations, whereas the broad peak at 3456 cm^{-1} was designated to O-H/N-H interactions. The interactions between the bonds are more extensive, and these bonds are indistinguishable. The peak identified at 810 cm^{-1} was ascribed to the C=C bending vibration. The presence of C-H stretching vibrations was confirmed by the peaks observed at 1038 and 2968 cm^{-1} . The identified peaks were correlated with the prior report [18].

The formation of CNC, phase, and average crystallite size were determined using XRD analysis. The XRD pattern of the synthesized CNC is presented in Figure 2. The prominent peaks were detected at 18.95° , 31.23° , 37.22° , 44.12° , 59.23° , and 65.25° , corresponding to the plane values (111), (220), (311), (400), (511), and (440), correspondingly, thereby affirming the synthesis of the CNC. The acquired XRD pattern corresponds with JCPDS card No. 73-1702, with a lattice parameter of $a = 8.11\text{ \AA}$. The space group is identified as

Fd3m, and the Bravais lattice is face-centered cubic (FCC). The synthesized CNC has a cubic spinel structure. In this scenario, Ni^{2+} generally occupies octahedral sites, Co^{3+} occupies both tetrahedral and octahedral sites, while the oxygen ions establish close-packed cubic arrangements. The peaks at 62.64° , 75.22° , and 79.15° may indicate the presence of CTAB or contaminants. Sivashanmugam et al. synthesized NiCo_2O_4 in an amorphous state, and the inclusion of CTAB transforms the amorphous structure into crystalline phases [19]. The average crystallite size was found by using the Debye–Scherrer equation (Equation (2)), which was calculated to be 18.56 nm [20].

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (2)$$

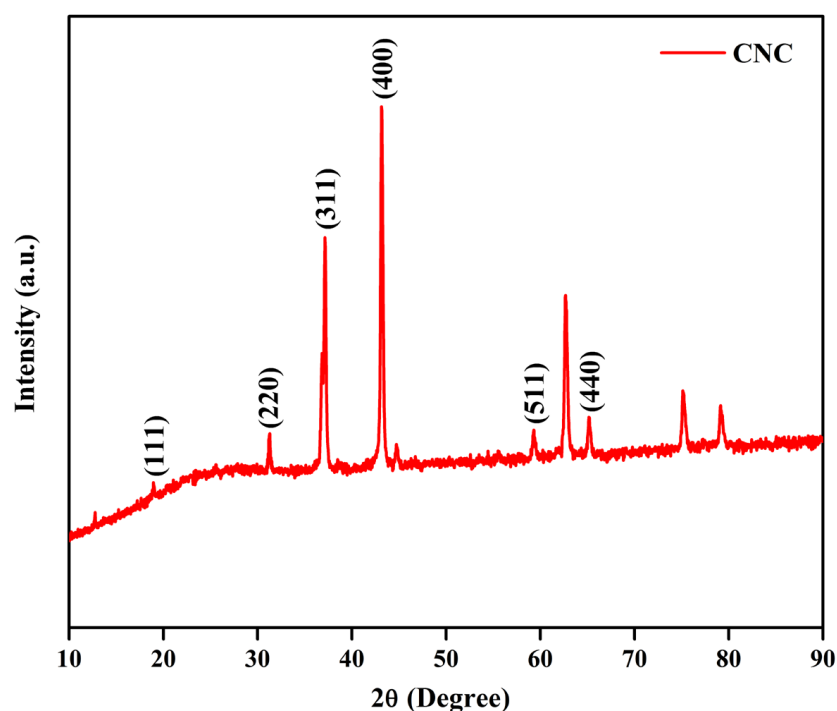


Figure 2. XRD pattern of the CNC.

The morphology and average particle size were assessed using TEM analysis, as illustrated in Figure 3a–d. The TEM pictures of CNC exhibited irregular spherical morphologies. The mean particle size of the CNC was determined to be roughly 160 nm using ImageJ software, Java (TM) Platform, Version 6. The images confirm that the CTAB template modifies the size and morphologies of the NiCo_2O_4 crystals. The primary function of CTAB is delineated as follows: CTAB is a cationic surfactant that fully ionizes in a solvent. The resulting cation is likewise a tetrahedron featuring an elongated hydrophobic tail. Due to electrostatic interactions, ion pairs may form between the metal ion and the CTA^+ cation. The absorbed metal ion from the surfactant serves as a crystal nucleus and a growth director for the formation of NiCo_2O_4 crystals. The CTAB template safeguards the aggregated structure; consequently, NiCo_2O_4 crystals are generated [21]. The presence of elements such as Ni, Co, O, and C in the CNC is confirmed by EDAX analysis. Additionally, Cu exists as an impurity in the CNC. Figure 3e illustrates the EDAX of CNC.

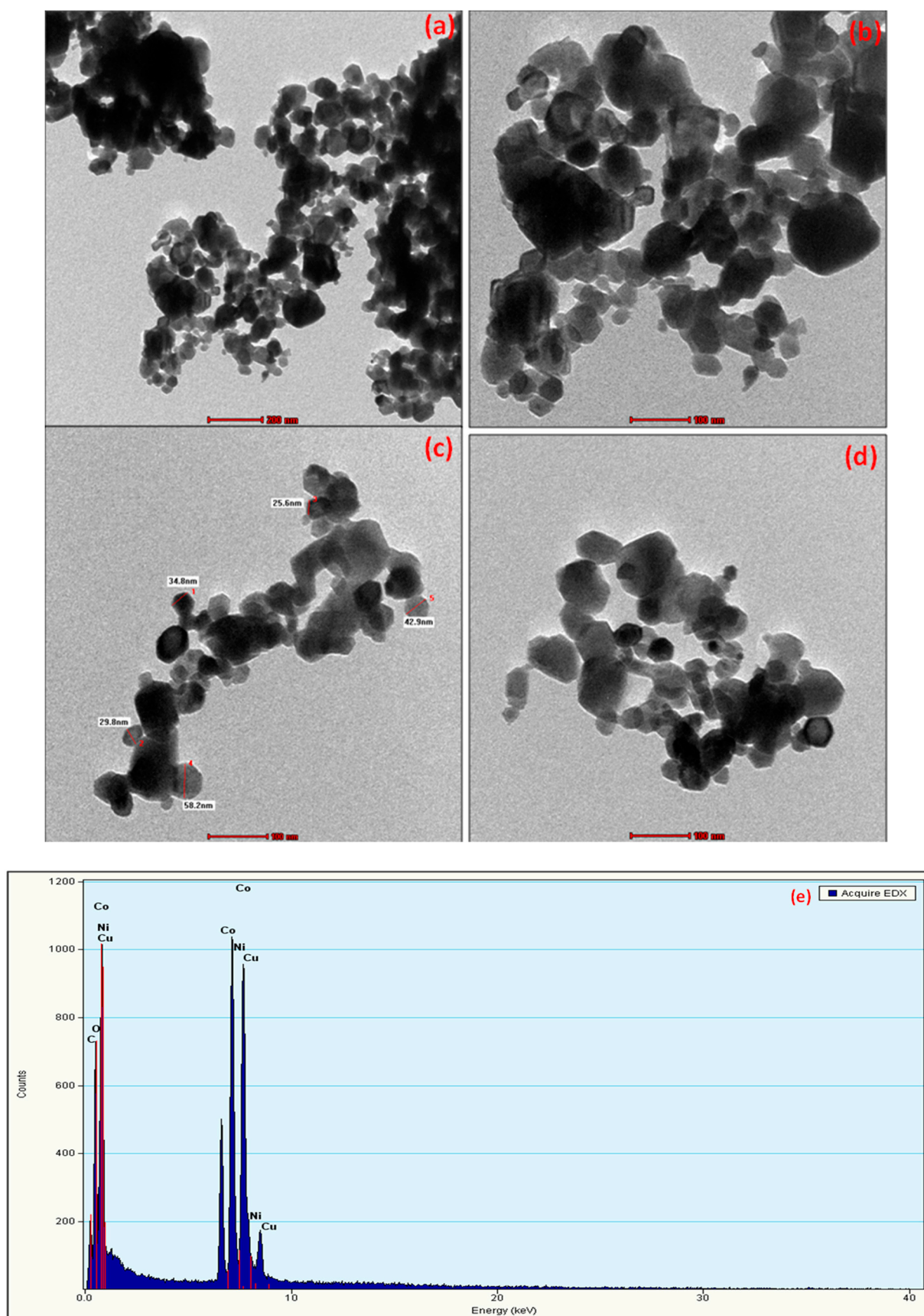


Figure 3. (a–d) TEM micrograph of CNC and (e) EDAX profile of the CNC.

2.2. Photocatalytic Degradation of the MG

The photocatalytic degradation of MG was conducted at optimized conditions of 10 ppm of MG concentration, pH 9, and 50 mg of catalyst weight. The photocatalytic activity was initiated by radicals generated during the degradation process by the interaction of excited electrons with water and dissolved molecules, which are responsible for commencing the destruction of contaminants. The deterioration efficiency was determined to be 97.62% under optimized conditions. Figure 4a illustrates the photocatalytic breakdown of malachite green when exposed to sunshine.

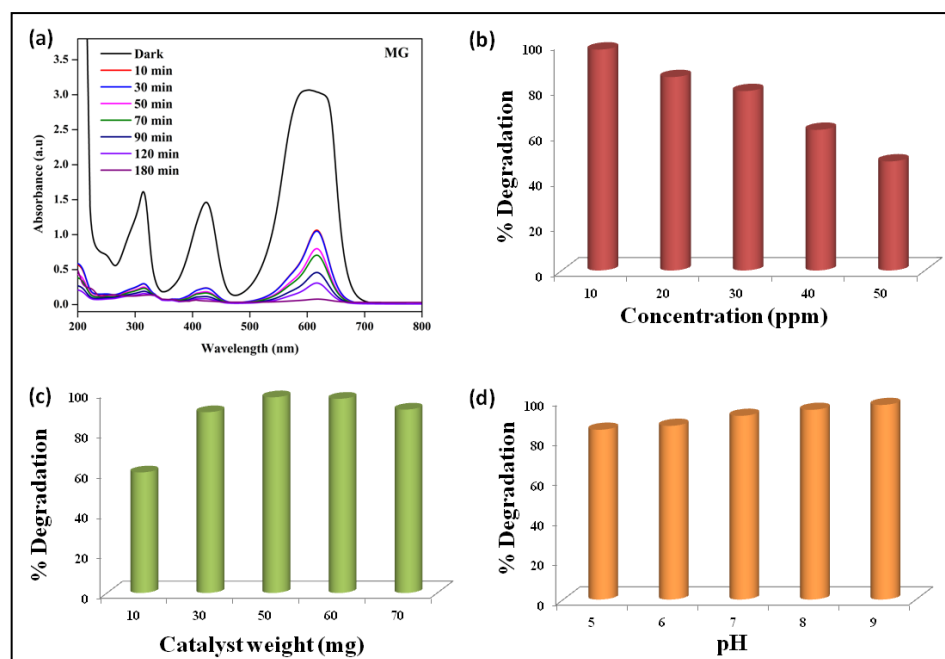


Figure 4. (a) Photocatalytic degradation of MG, (b) degradation efficiency at different concentrations, (c) degradation efficiency at different catalyst weights, and (d) degradation efficiency at different pH levels.

2.2.1. Effect of Concentration

The photocatalytic degradation of MG dye was investigated using CNC under solar light irradiation, varying the initial dye concentrations to 10, 20, 30, 40, and 50 ppm at $\text{pH} = 9 \pm 0.2$ and 50 mg of catalyst. The deterioration of MG was first recognized by a change in color, and it was diminished after 180 min of irradiation during photocatalysis. Subsequently, the deteriorated solution underwent UV-Vis analysis to ascertain the absorbance values. Throughout this process, the absorbance value progressively decreased as the duration of degradation increased. The degradation was achieved up to 97.62, 85.46, 79.27, 62.14, and 48.19% at 10, 20, 30, 40, and 50 ppm of MG, respectively. This is attributable to the accumulation of MG on the CNC with an increase in dye concentration. Figure 4a depicts the photocatalytic degradation of MG at different time intervals. The effectiveness of photocatalytic degradation of MG diminished as the initial concentration of the dye increased. Figure 4b illustrates the percentage degradation of MG as the dye concentration varies. The degradation efficiency reduced as the MG concentration was increased. It might be due to the decrease in the generation of electron–hole pairs and an increase in the intensity of the MG solution’s coloration at higher concentrations, which reduces light penetration owing to the shielding effect. Also, when there are too many dye molecules, the catalyst surface’s limited active sites become saturated; thus, many of them remain undegraded.

2.2.2. Effect of Catalyst Weight

The influence of catalyst weight on the degradation efficacy of photocatalysis concerning MG dye was examined by administering 10, 30, 50, 60, and 70 mg of CNC, while sustaining a constant dye concentration of 10 ppm and a pH of 9. The degradation efficiency of CNC against MG with different catalyst weights indicates that the maximum degradation percentage of 97.62% was achieved at 50 mg. Hence, the effectiveness of photocatalytic degradation improved with an increased catalyst dosage, as the dye molecules on the catalyst surface absorbed more available photons and radicals. This may result from an augmentation in the quantity of active sites inside the catalysts, hence enhancing the generation of hydroxyl radicals [22]. The generation of hydroxyl radicals also affected the catalyst's degrading efficiency. An increase in the catalyst weight of about 60 mg decreased the degradation efficiency. It may be due to the influence of the adsorption of active molecules on the surface of the catalyst, which prevented the excitation of photons in the CNC catalyst surface. Figure 4c depicts the degrading efficiency of the CNC as a function of the catalyst weight variation. The degradation efficiency was determined to be 60.14, 90.17, 97.62, 96.84, and 91.48% at 10, 30, 50, 60, and 70 mg of CNC.

2.2.3. Effect of pH

The comprehensive photocatalytic investigation is predominantly reliant on the influence of pH. The pH of the dye solution influences the photocatalytic degradation efficiency of the CNC catalyst. The impact of pH was assessed by adjusting the pH to 5, 6, 7, 8, and 9, utilizing HCl and NaOH. Figure 4d demonstrates the decreasing effectiveness of the CNC as a function of the pH change. The dye concentration was maintained at 10 ppm, and the irradiation duration was 180 min. The degradation efficiency was seen to be elevated at a high pH value of 9.0. The degradation percentages were determined to be 85.18, 87.14, 92.16, 95.17, and 97.62% at pH values of 5, 6, 7, 8, and 9, respectively. The degradation efficiency of MG was observed to improve at an alkaline pH due to the increased production of hydroxyl radicals, which are the principal oxidizing agents responsible for dye degradation. Furthermore, at elevated pH levels, the surfaces of the majority of metal oxides acquire a negative charge, hence enhancing electrostatic attraction and the destruction of cationic MG molecules. The enhanced degradation, along with the increased reactive oxygen species, results in superior degradation efficiency in the basic pH relative to acidic or neutral conditions. The photocatalytic degradation of MG by CNC follows the pseudo-first-order reaction, and the rate constant was determined to be 0.0175 min^{-1} . The kinetic study for the photocatalytic degradation of malachite green using CNC is depicted in Figure S1 (ESI[†]). Taken together, the optimum degradation efficiency of 97.62% was achieved at 10 ppm of dye concentration, 50 mg of catalyst loading, and pH 9, highlighting the favorable role of higher catalyst dosage and alkaline conditions, while higher dye concentrations suppressed photocatalytic activity. Table 1 depicts the comparison of the present work on the degradation of pollutants with the literature.

An investigation on reusability conducted for MG deterioration assesses the efficiency of the CNC across several usage cycles (Figure 5). This guarantees the CNC's stability, preserves its functionality, and prevents substantial degradation over time. Prior to commencing the reusability investigation, the CNC has been extracted from the reaction media and subsequently filtered and centrifuged. The catalyst was meticulously washed with water or ethanol to eliminate adsorbed dye or byproducts. The retrieved catalyst was dehydrated at 60°C in a convection oven. Subsequently, the recovered CNC was reintroduced into a new MG solution, and the degradation process was repeated under identical circumstances as the first cycle, with the degradation percentage deter-

mined after each cycle. The degradation efficiency decreased from 97.62% to 60.59% after the fifth cycle.

Table 1. Comparison of the results of degradation of pollutants with different catalysts.

S. No	Material	Pollutant	Efficiency and Time	Ref.
1	Cu- and Fe-doped NiCo ₂ O ₄ /g-C ₃ N ₄	Rhodamine B and Congo Red	89% and 120 min	[23]
2	carboxymethyl cellulose/ β -cyclodextrin/NiCo ₂ O ₄	Bisphenol-A Malachite Green Congo Red	<40% and 170 min 90% and 170 min No adsorption	[24]
3	NiCo ₂ O ₄ @CdS–Ag	Ofloxacin	99.14%	[25]
4	NiCo ₂ O ₄ /NiO	Rhodamine B	83.66% and 120 min	[26]
5	NiO/NiCo ₂ O ₄	Rhodamine B and Methylene Blue	94.75% and 120 min 93.55% and 120 min	[27]
6	CNC	Malachite Green	97.62% and 180 min	Present Work

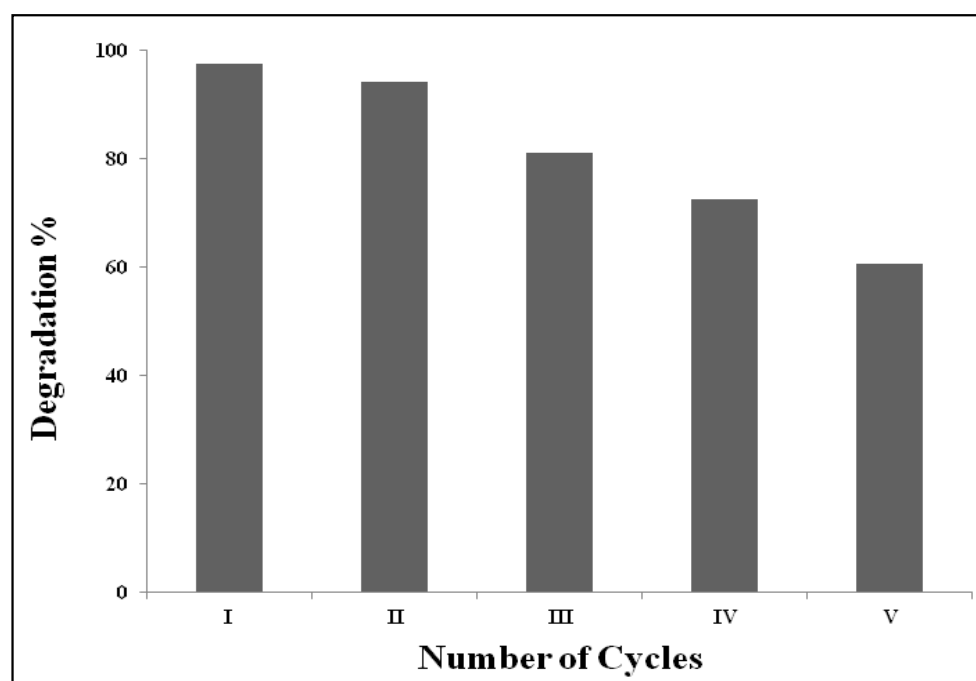


Figure 5. Reusability study of the degradation of malachite green using a catalyst.

The improved photocatalytic ability of the CNC can be attributed to the efficient movement of photoinduced electrons in CNC from the valence band to the conduction band, aided by a smaller band gap. These electrons accumulate on the CNC and are captured by dissolved oxygen to form superoxide anion radicals ($\bullet\text{O}_2^-$) and hydroxyl radicals ($\text{HO}\bullet$). Similarly, the photoinduced holes on the CNC react with adsorbed hydroxyl ions to produce additional hydroxyl radicals. These photoinduced electrons and holes generate reactive species that effectively break down and mineralize organic pollutants [1]. The degradation pathway of malachite green, illustrated in Figure 6, shows how these reactive hydroxyl radicals and superoxide anions transform into mineralized products once the reaction is complete (Figure 6).

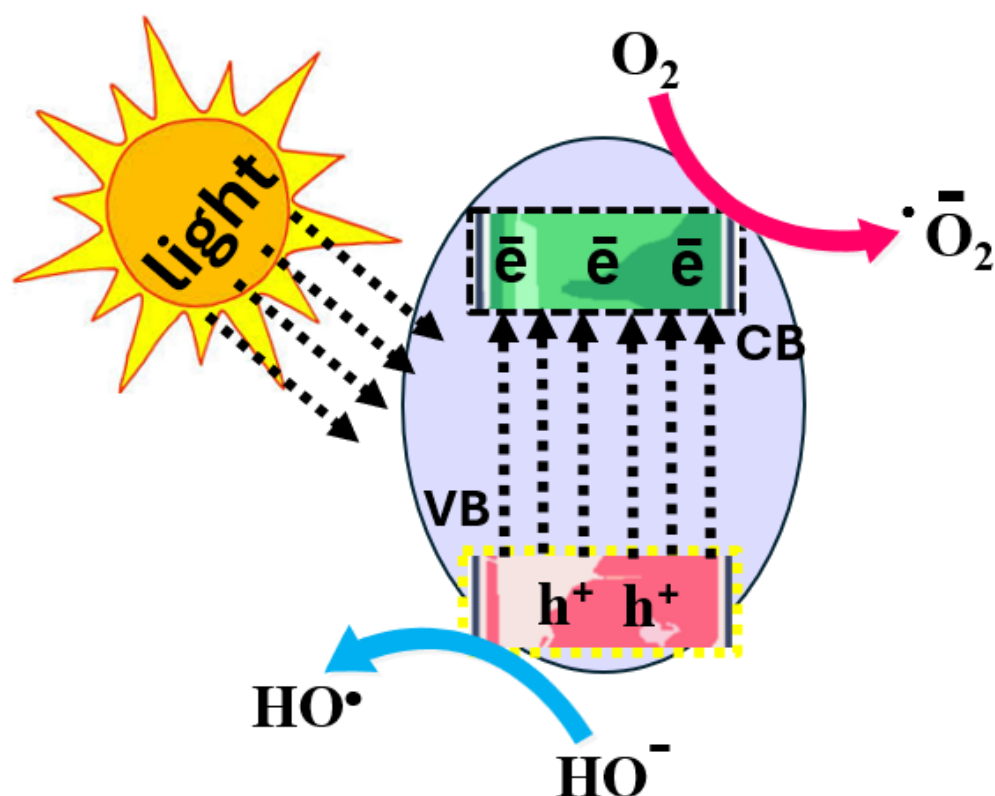


Figure 6. Proposed mechanism for the degradation of the MG over CNC.

2.3. Glucose Sensing Performance of the CNC

Figure 7a represents the CV analysis at different scan rates in the range of (10, 20, 30, 40, and 50 mV/s). In this study, the synthesized CNC shows excellent electrocatalytic performance, which suggests great potential for glucose detection. The anodic and cathodic peak currents increase with the scan rate, which indicates controlled redox processes. It is clearly observed that the anodic peak current increases from approximately 15 μ A at 10 mV/s to around 45 μ A at 50 mV/s. The peak separation (ΔE_p) increased from ~0.20 V at 10 mV/s to ~0.25 V at 50 mV/s. These values are significantly larger than the ~59 mV/n expected for an ideally reversible system, indicating that the process is more accurately described as quasi-reversible with predominantly diffusion-controlled behavior. This interpretation is further supported by the linear relationship between the peak current and the square root of the scan rate (Figure 7a). In Figure 7a, CV profiles recorded at different scan rates exhibit increasing current responses without distinct, sharp peak features. By contrast, in Figure 7b, the anodic peak is observed near +0.2 V and the cathodic peak is observed near −0.4 V, corresponding to the oxidation and reduction in the active species during glucose sensing. Based on these characteristics, it is clearly observed that the material shows efficient electrochemical activity, with a linear relationship between the peak current and the square root of the scan rate, which demonstrates diffusion-controlled kinetics. The observed current response correlated with the increasing concentrations of glucose, highlighting the material's sensitivity. This behavior can be attributed to the high surface area and active sites provided by the nanomaterials, which enhance glucose oxidation reactions [28].

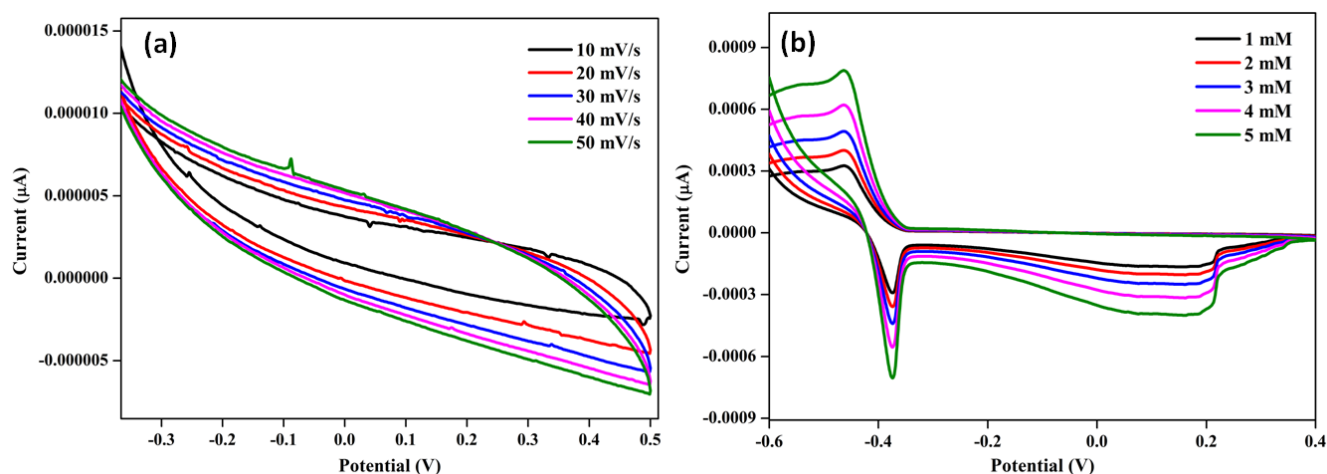


Figure 7. (a) CV profile of the CNC at different scan rates and (b) CV graph of the CNC with glucose at different concentrations.

The CV analysis shows the electrochemical response of the material to increasing glucose concentrations ranging from 1 mM to 5 mM at a constant scan rate. The results clearly show an increase in anodic and cathodic peak currents with an increasing glucose concentration. For instance, the cathodic peak current grows from approximately -5×10^{-5} A at 1 mM to about -2.5×10^{-4} A at 5 mM (Figure 7b). Similarly, the anodic peak current increases from around 0.0001 A at 1 mM to approximately 3×10^{-4} A at 5 mM. In Figure 7b, the peak potentials remain consistent across glucose concentrations, with the anodic peak observed near +0.2 V and the cathodic peak near -0.4 V, signifying stable electrode kinetics. The results show that the synthesized CNCs were employed as electrodes for non-enzymatic glucose detection. The sensor's high sensitivity can be attributed to the synergistic effects of nickel and cobalt ions within the spinel structure, which facilitate glucose oxidation and reduce overpotentials. It should be emphasized that the present glucose sensing results are preliminary and reported per geometric electrode area. Full analytical validation, including calibration plots with LOD/LOQ, reproducibility, selectivity, drift/stability, and real-sample testing, along with ECSA-normalized activities, will be addressed in future work.

Table 2 represents the comparison result of the detection of glucose with different catalysts. Figure S2 (ESI†) shows the Nyquist plot AC impedance analysis of the material, which shows the relationship between the real part (Z') and the imaginary part (Z'') of the impedance. The semicircular region at lower Z' values (~ 0 –100 Ω) indicates the charge transfer resistance (R_{ct}), corresponding to the electrochemical reaction at the electrode interface. The linear portion observed at higher Z' values (~ 100 –500 Ω) represents the diffusion-controlled Warburg impedance. The maximum imaginary impedance (Z'') reaches approximately -800Ω , with the real impedance (Z') extending up to 500 Ω . The observed behavior clearly shows the highlights of the material's efficient charge transport and catalytic properties, confirming its suitability for sensor applications. The achieved results can be attributed to the uniform distribution of active sites and the porous structure of the NiCo_2O_4 material, which provide ion diffusion and improve the electron transfer rate. The results align with morphological and compositional characterizations, confirming the material's ability to interact effectively with glucose molecules.

Table 2. Comparison result of detection of glucose with different catalysts.

S. No	Catalyst	Sample	Efficiency	Ref.
1	Graphene/Co ₃ O ₄	Enzyme-free glucose	214 $\mu\text{A mM}^{-1} \text{cm}^{-2}$ at 0.49 s	[29]
2	Co ₃ O ₄	Glucose in saliva	2495.79 $\mu\text{A mM}^{-1} \text{cm}^{-2}$	[30]
3	ZnO/Co ₃ O ₄ /Graphene oxide	Enzyme-free glucose	1551.38 $\mu\text{A mM}^{-1} \text{cm}^{-2}$ at 3 s	[31]
4	NiO nanoporous film	Enzyme-free glucose	1202 $\mu\text{A mM}^{-1} \text{cm}^{-2}$	[32]
5	CNC	Enzyme-free glucose	159 $\mu\text{A mM}^{-1} \text{cm}^{-2}$ and LOD $\sim 0.38 \text{ mM}$	Present Work

3. Materials and Methods

3.1. Materials

All compounds, including nickel nitrate hexahydrate, cobalt nitrate hexahydrate, cetyltrimethylammonium bromide (CTAB), and ammonium hydroxide, were procured from Sigma Aldrich, St. Louis, MO, USA and utilized without additional purification. Malachite green was chosen as a contaminant for the catalytic study. Deionized water served as the solvent for the experimental procedure.

3.2. Fabrication of the CNC

A 1:1 ratio of metal precursors (1 mM of nickel nitrate hexahydrate and cobalt nitrate hexahydrate) was diluted in 30 mL of distilled water and agitated for 30 min to achieve equilibrium. Ammonium hydroxide was added dropwise to sustain a pH of 12 ± 0.2 , and stirring was maintained for 30 min. Subsequently, 10.9 mg of CTAB was introduced to the aforementioned reaction mixture and stirred for 30 min to modify the material's morphology. Subsequently, the synthesized bimetal hydroxide was rinsed with deionized water and ethanol to remove unreacted precursor materials and impurities, then dried at 80 °C in a hot air oven and further annealed at 600 °C to convert the hydroxide into oxide material. The produced CNC was stored in an airtight container for eventual analytical and application uses.

3.3. Methods

The absorption maximum was determined using a JASCO UV-Vis spectrophotometer, Tokyo, Japan. The IR spectrometer was employed to verify the functional groups and the synthesis of metal oxide, recorded in the wavenumber range of 4000–400 cm^{-1} using a JASCO IR spectrometer, Tokyo, Japan at a speed of 2 mm/s. The crystallite size, phase, and structure were determined by X-ray diffraction (XRD) conducted with an XPERT-PRO diffractometer (Philips Analysis Instruments, Amsterdam, The Netherlands) employing Cu K α radiation ($\lambda = 1.540 \text{ \AA}$, 40 kV, 15 mA). The XRD pattern was conducted between 10 and 90 degrees. Morphology was determined using a Tecnai G2, 120 kVA, FEI Company (Hillsboro, OR, USA) transmission electron microscope, and the elements present in the composite were analyzed with EDAX at an operating voltage of 15 kV. The EDX analysis was employed to determine the atomic and weight percentages.

3.4. Photocatalytic Degradation of Malachite Green

The photocatalytic degradation of MG was conducted using sunlight as the irradiation source. The standard approach involves a reaction vessel containing 250 mL of a 10 ppm MG solution and 50 mg of catalyst, with the pH maintained at 9 ± 0.2 . The reaction was conducted in darkness for the initial 30 min to achieve adsorption–desorption on the catalyst's surface. Subsequently, the reaction vessel was positioned in sunlight, and

the intensity of the sunlight was continuously measured (830–850 lux). Subsequently, the reaction commenced, and the deteriorated MG was collected at particular time intervals for a duration of 3 h. The absorption value of MG was recorded at 617 nm using a Shimadzu UV–visible spectrophotometer model 2600, Kyoto, Japan. The deterioration percentage was determined using the formula presented in Equation (3).

$$\% \text{ Degradation} = \frac{C_0 - C_t}{C_0} \times 100 \quad (3)$$

where C_0 is the concentration of the MG before degradation and C_t is the concentration of the MG at 't' time.

3.5. Electrochemical Measurements

Electrochemical measurements were conducted for the glucose sensor application at ambient temperature. The standard three-electrode system (working electrode-prepared carbon paste electrode with CNC, reference electrode Ag/AgCl electrode, and indicator electrode-platinum wire) was used for the electrochemical tests. The glassy carbon electrode (GCE) was initially polished and subjected to ultrasonic cleaning. The CNC catalyst was suspended in ethanol with 0.05% Nafion binder. Then, the catalyst was coated on the GCE by the drop-cast method, and it was air-dried. Finally, the modified electrode was employed for electrochemical glucose detection. The 1 M KOH solution was used as an electrolyte, and D-glucose was used as an analyte for the glucose sensor study [33].

4. Conclusions

In this study, the CNC was synthesized using a straightforward and cost-effective co-precipitation method and was tested for its utility in environmental and biosensing applications. Detailed analysis confirmed that the CNC has a cubic spinel structure and exhibits semiconductor properties, making it suitable for photocatalytic and electrocatalytic applications. The band gap measured by UV–visible spectroscopy was 2.15 eV, confirming the CNC's semiconductor nature. Additionally, the presence of metal oxide bonds in the CNC was confirmed by a distinct peak at 667 cm^{-1} observed in the FTIR spectrum. Further examination revealed the CNC's nanorod and irregular spherical shapes with an average particle size of 160 nm and a nanorod width of about 30 nm. The average crystallite size was determined to be around 18 nm. These structural characteristics contribute to its high reactivity and functional performance. The synthesized CNC showed excellent performance in removing the malachite green dye from solutions, achieving a degradation efficiency of 97% under optimal conditions. This high level of effectiveness indicates its potential for use in water purification processes. Additionally, the CNC proved capable in the electrochemical detection of glucose, showing an increase in the electrical current with higher glucose concentrations, which points to its effectiveness as a biosensor. The cathodic peak current increased from roughly $-5 \times 10^{-5} \text{ A}$ at a 1 mM glucose concentration to about $-2.5 \times 10^{-4} \text{ A}$ at 5 mM. Similarly, the anodic peak current rose from approximately $1 \times 10^{-4} \text{ A}$ at 1 mM to about $3 \times 10^{-4} \text{ A}$ at 5 mM. The stability of the electrode reactions was highlighted by the consistent peak potentials, with the anodic peak near 0.25 V and the cathodic peak around -0.3 V . This stability and the increased current responses indicate that CNC is a highly effective material for facilitating photoelectrochemical reactions. Overall, the results from this research confirm that CNC is a powerful catalyst that can be used both for breaking down environmental pollutants and for monitoring glucose levels, showcasing its versatility and potential for broader applications in environmental cleanup and health monitoring technologies.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/catal15090899/s1>, Figure S1: Pseudo-first order kinetics for the degradation of malachite green; Figure S2: AC Impedance in glucose detection.

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