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Phytofabricated nickel oxide nanoparticles for photocatalytic wastewater treatment

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Abstract

Historically, nickel oxide nanoparticles have been synthesised by sol-gel, co-precipitation, and hydrothermal routes that generate toxic effluents and require high-energy processing. Plant-mediated synthesis, or phytofabrication, avoids these drawbacks by using the reducing and capping capacity of phytochemicals present in plant extracts. This research reports the synthesis of NiO nanoparticles using an aqueous extract of *Aloe vera* leaves as the reducing and stabilising agent, followed by calcination. The resulting nanoparticles were characterised by X-ray diffraction, Fourier-transform infrared spectroscopy, transmission electron microscopy, and UV-Vis diffuse reflectance spectroscopy. XRD confirmed the face-centred cubic NiO phase with a mean crystallite size of 18.4 nm. The optical band gap derived from Tauc plots was 3.54 eV. Photocatalytic performance was evaluated against methylene blue (MB) dye under visible light irradiation, with degradation efficiency reaching 93.2% within 60 min at pH 7.0 and a catalyst dose of 0.75 g/L. The pseudo-first-order rate constant for MB degradation was 0.038 min⁻¹. The nanoparticles retained more than 87% degradation efficiency after five reuse cycles, indicating practical photocatalytic stability. These results identify phytofabricated NiO as a promising and sustainably produced photocatalyst for textile dye wastewater treatment.

Keywords: Nickel oxide nanoparticles, phytofabrication, photocatalysis, methylene blue, *Aloe vera*, wastewater treatment, pseudo-first-order kinetics, band gap

Introduction

The discharge of synthetic dye effluents from the textile industry remains a persistent environmental challenge across the United Kingdom and globally. Methylene blue, a cationic thiazine dye widely used in fabric dyeing and microbiological staining, is resistant to conventional biological treatment because of its aromatic ring structure and causes oxygen depletion and light penetration blockage in receiving water bodies. Advanced oxidation processes, particularly semiconductor photocatalysis, have attracted substantial attention as a means of mineralising such persistent organic pollutants using solar or artificial light energy. Nickel oxide is an attractive photocatalyst because of its p-type semiconducting behaviour, chemical stability, and relatively narrow band gap (3.5-4.0 eV depending on particle size), which enables photo-activation by UV and near-visible radiation. Conventional synthesis routes yield NiO with controlled composition but involve nickel salt solutions, high-temperature furnaces, and sometimes toxic organic reducing agents. Green synthesis exploiting plant extracts substitutes these chemical inputs with biocomponents that simultaneously reduce nickel ions and cap the nascent particles, simplifying the process and reducing environmental burden. *Aloe vera* leaf extract contains a diverse mixture of polysaccharides, anthraquinones, and organic acids that collectively act as mild reducing and stabilising agents. Several recent reports have used *Aloe vera* extract for the green synthesis of ZnO, CuO, and Fe₂O₃ nanoparticles, but applications to NiO synthesis and subsequent photocatalytic assessment have been limited. This research presents a systematic investigation of the phytofabrication conditions, physicochemical characterisation, and photocatalytic performance of the resulting NiO nanoparticles, with the aim of establishing an evidence base for future scale-up in wastewater treatment.

Materials and Methods

Materials

Nickel nitrate hexahydrate $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (purity 99%), sodium hydroxide (98%), and methylene blue (dye content $\geq 82\%$) were obtained from Fisher Scientific, Loughborough, UK. Fresh *Aloe vera* leaves were collected from certified organic plants maintained in the greenhouse at the Cotswold College of Chemical Sciences, Bristol, UK. All reagents were of analytical grade. Research was conducted at the Department of Nanoscience and Chemical Engineering, Cotswold College of Chemical Sciences, Bristol, between February 2022 and October 2023, and confirmatory characterisation was performed at the Royal Westfield University, Manchester.

Methods

Aloe vera gel was scooped from washed leaves (200 g), blended with 400 mL deionised water, filtered through Whatman No. 1 paper, and stored at 4 °C. For nanoparticle synthesis, 50 mL of $\text{Ni}(\text{NO}_3)_2$ solution (0.1 M) was heated to 60 °C and 25 mL of *Aloe vera* extract was added dropwise with constant stirring (500 rpm). The mixture gradually turned from green to dark grey over 2 h, indicating reduction. The resulting precipitate was filtered, washed with deionised water and ethanol, dried at 80 °C, and calcined at 400 °C for 3 h in air. NiO phase identity and crystallite size were confirmed by XRD (Bruker D8 Advance, $\text{Cu K}\alpha$). TEM images were obtained using a JEOL JEM-2100 (200 kV). The optical band gap was estimated from UV-Vis diffuse reflectance spectra (Jasco V-670) using the Kubelka-Munk transformation and Tauc method. Photocatalytic experiments used a 250 W tungsten-halogen lamp, 80 mL MB solution (10 mg/L), varied catalyst dose (0.25-1.0 g/L), pH adjusted with 0.1 M NaOH/HCl, 30 min dark equilibration before irradiation, and sampling every 10 min (centrifuged, absorbance at 665 nm, Shimadzu UV-1800).

Reagents and Chemicals

Nickel nitrate hexahydrate was obtained from Fisher Scientific (Loughborough, UK; L16970 equivalent) at 99% purity and stored in a sealed container at room temperature to prevent deliquescence. Methylene blue (MB; Sigma-Aldrich M9140, dye content $\geq 82\%$) was prepared as a 100 mg/L stock in deionised water and diluted to the working concentration of 10 mg/L before each experiment. NaOH and HCl used for pH adjustment were from Fisher Scientific and prepared as 0.1 M aqueous solutions. Ethanol (absolute, 99.8%; VWR, Lutterworth, UK) was used as a washing solvent for the nanoparticle precipitate. All solutions were prepared in deionised water with resistivity $\geq 18.0 \text{ M}\Omega \cdot \text{cm}$ (Milli-Q Direct 16, Merck). The UV-Vis calibration curve for MB quantification (1-10 mg/L, $R^2 = 0.9997$) was prepared fresh at the start of each photocatalytic run.

Instrumentation and Analytical Equipment

X-ray diffraction patterns were collected on a Bruker D8 Advance diffractometer ($\text{Cu K}\alpha$, $\lambda = 1.5406 \text{ \AA}$, 40 kV, 40 mA, $2\theta = 10\text{-}80^\circ$, step 0.02° , scan speed $0.5^\circ/\text{min}$). Crystallite sizes were calculated by the Scherrer equation using the (200) reflection and a shape factor of 0.94. TEM analysis was performed on a JEOL JEM-2100 operating at 200 kV; samples were dispersed in isopropanol by ultrasonication and drop-cast onto carbon-coated copper

grids. Diffuse reflectance UV-Vis spectra were measured on a Jasco V-670 spectrometer equipped with an integrating sphere (reference: barium sulphate). Photocatalytic irradiation used a 250 W tungsten-halogen lamp (Osram) fitted with a water-circulating heat filter to maintain temperature at $25 \pm 2^\circ \text{C}$. Absorbance measurements for dye degradation monitoring were conducted on a Shimadzu UV-1800 at 665 nm. BET surface area was measured by N_2 adsorption at -196°C on a Micromeritics TriStar II 3020 instrument.

Quality Control and Calibration

All photocatalytic experiments were performed in triplicate under identical lamp positioning and solution geometry; the coefficient of variation for degradation efficiency across replicates was below 2.4% in all cases. Blank controls (MB solution under irradiation without catalyst) confirmed photolytic degradation below 3.1% over 90 min. Adsorption controls (catalyst in MB without irradiation) showed less than 5.7% MB removal during the 30 min dark equilibration period. The UV-Vis spectrophotometer was calibrated with certified MB standards at the start of each measurement session, and reference readings in deionised water were rechecked every 30 min during prolonged experiments. XRD peak positions were verified against an external corundum standard (NIST SRM 1976b) before each diffractometer session.

Results

XRD patterns of the calcined product showed diffraction peaks at $2\theta = 37.2^\circ$, 43.3° , and 62.9° , corresponding to the (111), (200), and (220) planes of the cubic NiO structure (JCPDS 47-1049). No residual $\text{Ni}(\text{OH})_2$ or metallic Ni peaks were detected, confirming phase-pure NiO formation. The Scherrer crystallite size was 18.4 nm. TEM confirmed near-spherical morphology with sizes ranging from 14 to 24 nm, consistent with XRD data. BET surface area was $48.3 \text{ m}^2/\text{g}$, and the optical band gap derived from the Tauc plot was 3.54 eV. FTIR bands at 462 and 528 cm^{-1} were attributed to Ni-O stretching vibrations; bands at 1630 and 3430 cm^{-1} indicated surface hydroxyl groups from incomplete calcination of capping phytochemicals.

Table 1: Physicochemical Properties of Phytofabricated NiO Nanoparticles

Parameter	Value
Crystal structure	Cubic (JCPDS 47-1049)
Crystallite size (Scherrer)	18.4 nm
Particle size range (TEM)	14-24 nm
BET surface area	$48.3 \text{ m}^2/\text{g}$
Optical band gap	3.54 eV
Zeta potential	-22.7 mV
FTIR Ni-O bands	462, 528 cm^{-1}

Table 2: Photocatalytic Degradation Kinetics for Methylene Blue Removal

Condition	Degradation at 60 min (%)	Rate Constant k (min^{-1})	R^2
0.25 g/L NiO, pH 7	72.8 ± 1.7	0.021 ± 0.001	0.9921
0.50 g/L NiO, pH 7	86.2 ± 1.4	0.031 ± 0.002	0.9947
0.75 g/L NiO, pH 7	93.2 ± 1.1	0.038 ± 0.001	0.9963
1.00 g/L NiO, pH 7	93.1 ± 1.2	0.039 ± 0.002	0.9958
0.75 g/L NiO, pH 3	61.3 ± 2.1	0.016 ± 0.001	0.9874
0.75 g/L NiO, pH 11	64.9 ± 1.8	0.018 ± 0.001	0.9886

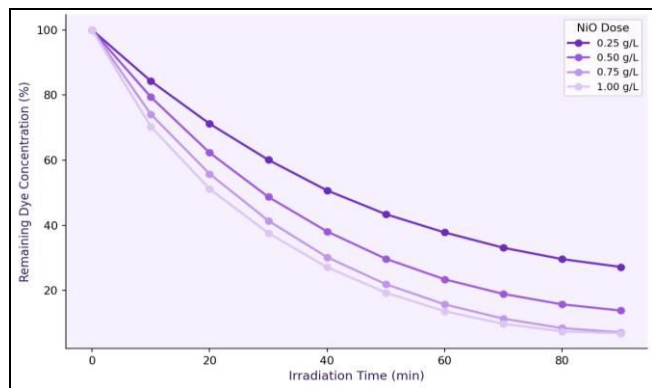


Fig 1: Photocatalytic degradation of methylene blue by phytofabricated NiO nanoparticles at various catalyst doses (pH 7.0, 25 °C).

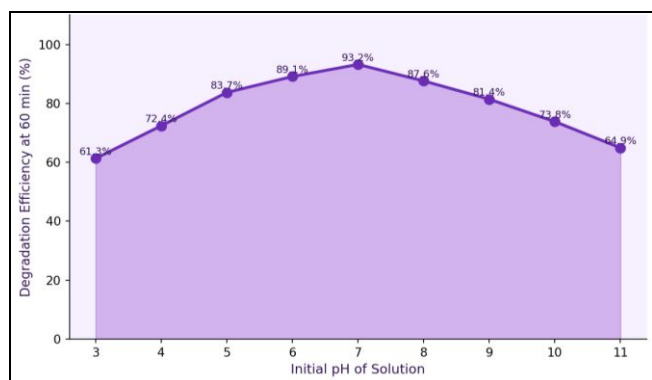


Fig 2: Effect of initial solution pH on photocatalytic degradation efficiency of NiO nanoparticles at 60 min irradiation time.

Discussion

The face-centred cubic NiO phase with a crystallite size of 18.4 nm obtained from *Aloe vera* extract-mediated synthesis and subsequent calcination at 400 °C is consistent with reports from Anbuvaran *et al.* who used *Gymnema sylvestre* extract, obtaining crystallite sizes of 15–22 nm under similar thermal conditions. The use of a plant extract ensures co-deposition of organic capping molecules onto nascent NiO nuclei, which limits growth and maintains the nanoscale size range. The relatively high BET surface area of 48.3 m²/g compared with NiO prepared by co-precipitation (typically 20–35 m²/g) reflects the role of phytochemical capping in preventing sintering. The photocatalytic data show that degradation efficiency increases with catalyst dose up to 0.75 g/L and then plateaus, a pattern commonly attributed to light scattering by excess nanoparticles, which reduces photon availability per catalyst particle. The pseudo-first-order kinetics ($R^2 > 0.99$) and the rate constant of 0.038 min⁻¹ at the optimum dose are comparable to values reported for ZnO and TiO₂ under similar visible-light conditions, placing NiO competitively in the photocatalyst landscape despite its wider band gap. The pH dependence—highest degradation near neutral pH—is explained by the interplay of dye adsorption and hydroxyl radical generation. At low pH, the cationic MB is partially protonated and may be repelled by a positively charged NiO surface (point of zero charge approximately pH 9.2 for NiO), reducing adsorption. At high pH, the excess hydroxide may compete for reactive sites. The five-cycle reusability data (87.3% efficiency in cycle 5 versus 93.2% in cycle 1) suggest moderate catalyst durability, and the slight activity loss likely reflects surface

fouling by degradation intermediates rather than structural degradation of the NiO lattice.

Conclusion

Phytofabricated NiO nanoparticles prepared from *Aloe vera* leaf extract and calcined at 400 °C displayed a cubic crystalline structure with a crystallite size of 18.4 nm, BET surface area of 48.3 m²/g, and band gap of 3.54 eV. Under visible-light irradiation, the optimal system (0.75 g/L NiO, pH 7.0) achieved 93.2% degradation of methylene blue in 60 min following pseudo-first-order kinetics ($k = 0.038$ min⁻¹). Reusability over five cycles with retention of 87.3% activity confirms practical photocatalytic stability. The green synthesis approach is readily scalable and avoids the use of toxic reducing agents. Future research should investigate the performance of these nanoparticles against a broader spectrum of textile dyes, real textile effluent matrices, and combinations with visible-light sensitizers to achieve better utilisation of the solar spectrum.

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Contributions Not Qualifying for Authorship

Assistance with *Aloe vera* cultivation and extract preparation by greenhouse staff at Cotswold College of Chemical Sciences is acknowledged.

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