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SYNTHESIS AND CHARACTERIZATIONS OF NICKEL OXIDE (NIO) NANOPARTICLES USING THE SOL-GEL METHOD

Abdul Basit (Corresponding Author)
Department of Chemistry, University of science
And Technology, Bannu, Pakistan.
Email: abdulbasitustb@gmail.com

Waqar Ahmad
Department of Chemistry, University of science
And Technology,Bannu, Pakistan.
Email: Waqarahmad7272kundi@gmail.com

Dr. Farman Ullah Khan
Assistant Professor Department of Chemistry, University
Of science And Technology Bannu, Pakistan.
dr.farmanullah@ustn.edu.pk
Email: farmandphil@yahoo.com

Waseem Ullah
Department of Chemistry, University of science
And Technology Bannu,Pakistan.
Email: udrwaseem@gmail.com

Nazish Noreen
Department of Chemistry, University of Science
And Technology, Bannu, Pakistan.
Email: nazishnoreen148@gmail.com

Sana saeed
Chemical Engineering Department, NFC Institute of
Engineering and Technology, Multan, Pakistan.
Email: sanasaeed@nfciet.edu.pk

Zafar Nawaz
Société Générale de Surveillance (SGS), Hayatabad, 25000 Peshawar, Khyber Pakhtunkhwa, Pakistan
Email: zafarnawaz2570@gmail.com

Muhammad Masood Zafar
Department of Chemistry, Division of Science and Technology, University of Education, Lahore,Panjab, Pakistan.
bsf1600806@ue.edu.pk
Email: masoodzafar52@gmail.com
https://orcid.org/0009-0005-5070-4465

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ABSTRACT

Nickel oxide (NiO) nanoparticles were effectively produced using a low-temperature sol–gel technique, utilizing sodium hydroxide in methanol as the precipitating agent and nickel sulfate hexahydrate as the precursor. The production process included gel formation and subsequent controlled calcination at 450 °C, resulting in fine, black NiO nanoparticles with homogeneous shape. The structural, chemical, and optical characteristics of the synthesized nanoparticles were examined utilizing Fourier Transform Infrared Spectroscopy (FTIR) and UV-visible spectroscopy. FTIR research verified the existence of distinctive Ni–O stretching vibrations, accompanied by small contributions from hydroxyl groups, indicating partial surface adsorption. UV–visible spectroscopy demonstrated a clear absorption edge at 328 nm, underscoring the material's semiconducting properties. These results validate the efficacy of the sol–gel method in synthesizing NiO nanoparticles with adjustable optical and structural characteristics. Due to these characteristics, the produced NiO nanoparticles exhibit significant potential for various applications in catalysis, optoelectronic devices, and environmental remediation technologies.

Keywords
Nickel oxide nanoparticles; Sol–gel synthesis; FTIR; UV–visible spectroscopy; Semiconducting properties; Catalysis; Optoelectronics; Environmental applications

Introduction

Nanotechnology has evolved as a revolutionary domain of science and engineering, facilitating the manipulation of matter at the nanoscale to create materials with distinctive and superior features relative to their bulk equivalents (Bhushan, 2017; Ahamd et al; 2023). Metal oxide nanoparticles have garnered significant interest among several types of nanomaterials due to their diverse structural, electrical, and optical properties (Schrofel et al., 2014; Ahamd et al 2024)). These materials have been thoroughly investigated for use in catalysis, sensors, energy storage, photocatalysis, and optoelectronics, as their diminished dimensions and elevated surface area-to-volume ratio markedly modify their physicochemical properties (Sharma et al., 2019; Mumtaz et al 2024; Ahmad et al., 2025). In this context, nickel oxide (NiO), a p-type semiconductor characterized by a broad band gap, emerges as one of the most promising transition metal oxides (Bonomo & Dini, 2016).

NiO is a binary oxide characterized by a rock-salt crystal structure and significant thermal and chemical stability (Kumar et al., 2015). This material is acknowledged for its strategic significance owing to its extensive band gap (3.6–4.0 eV in bulk form), elevated refractive index, and superior electrochemical characteristics (Chen et al., 2015). These characteristics render NiO nanoparticles exceptionally appealing for several advanced applications, such as electrochromic devices, gas sensors, transparent

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conductive films, energy storage electrodes, photocatalysis, and heterogeneous catalysis (Subramanian et al., 2014; Bonomo & Dini, 2016). The environmental compatibility and comparatively low cost of nickel precursors render NiO-based nanomaterials especially attractive for large-scale and sustainable technological applications (Kayani et al., 2018).

The characteristics of NiO nanoparticles are significantly influenced by their particle size, shape, crystallinity, and surface chemistry (Hu et al., 2012). At the nanoscale, NiO demonstrates quantum confinement effects that alter its optical absorption edge, consequently modifying its electronic and semiconducting properties (Xu et al., 2013). The prevalence of surface flaws and active sites in nanosized NiO enhances its catalytic activity relative to bulk materials (Alagiri et al., 2012). These characteristics highlight the significance of choosing appropriate synthesis methods to get regulated particle sizes and intended functions. Diverse chemical and physical methodologies have been utilized for the synthesis of NiO nanoparticles, including hydrothermal synthesis, co-precipitation, thermal decomposition, solvothermal procedures, and sol-gel processes (Kumar et al., 2015; Kayani et al., 2018). The sol–gel approach has become one of the most effective and adaptable techniques for synthesizing homogeneous NiO nanoparticles with regulated size and shape under relatively mild conditions (Alagiri et al., 2012).

The sol–gel technique is a solution-based technology that facilitates the transformation of a system from a colloidal suspension (sol) to a continuous solid network (gel). This approach provides numerous benefits, including low synthesis temperature, high product purity, compositional uniformity, and the ability to customize particle size and shape by modifying reaction parameters (Brinker & Scherer, 1990). The synthesis of NiO with sodium hydroxide as the precipitating agent and nickel salts as precursors offers an economical and direct method to produce nickel hydroxide gels, which, upon calcination, convert into crystalline NiO nanoparticles (Kayani et al., 2018). Calcination eliminates organic and residual hydroxyl groups while boosting crystallinity, therefore improving the material's optical and structural properties (Hu et al., 2012).

The characterization of NiO nanoparticles is essential for comprehending their structural and functional properties. Fourier Transform Infrared Spectroscopy (FTIR) is frequently utilized to identify chemical bonds, verify Ni–O connections, and discover residual hydroxyl groups or surface-adsorbed species (Chen et al., 2015). UV–visible spectroscopy, conversely, offers significant insights into the optical properties and band gap of the produced nanoparticles (Xu et al., 2013). The presence of a unique absorption edge in the UV spectrum signifies the semiconducting characteristics of NiO, which are crucial for optoelectronic and photocatalytic applications. By combining synthesis circumstances with spectroscopic analysis, researchers can delineate dependable methodologies to fabricate NiO nanoparticles with specified properties designed for targeted applications (Sharma et al., 2019).

The technological capabilities of NiO nanoparticles encompass a wide array of fields. In catalysis, NiO has exhibited significant efficacy in oxidation-reduction reactions, pollutant degradation, and hydrogenation processes (Subramanian et al., 2014). Its elevated surface reactivity and capacity to offer active sites render it an effective

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catalyst in environmental cleanup technologies (Bonomo & Dini, 2016). In optoelectronics, NiO functions as a hole transport layer in solar cells and light-emitting diodes (LEDs) owing to its broad band gap and p-type semiconducting characteristics (Chen et al., 2015). Moreover, NiO nanoparticles are progressively utilized in electrochemical energy storage devices, including supercapacitors and lithium-ion batteries, due to their redox activity, which improves charge storage capacity and cycling stability (Kumar et al., 2015; Sharma et al., 2019). NiO's versatility encompasses biomedical applications, where it has been explored for antibacterial and anticancer properties; nevertheless, these applications necessitate meticulous assessment of biocompatibility (Hu et al., 2012).

Notwithstanding these benefits, obstacles persist in enhancing the production and processing of NiO nanoparticles for practical applications. Regulation of particle size distribution, inhibition of agglomeration, and precise adjustment of surface chemistry are critical factors influencing performance (Xu et al., 2013). The sol–gel method offers a strong framework to address these constraints by facilitating molecular-level regulation of the reaction environment. The gentle synthesis conditions and versatility of this process facilitate the generation of high-quality NiO nanoparticles appropriate for both fundamental research and industrial application (Alagiri et al., 2012; Kayani et al., 2018).

This study generated NiO nanoparticles by a low-temperature sol–gel method, utilizing sodium hydroxide as the precipitating agent in methanol and nickel sulfate hexahydrate as the precursor. The resultant material underwent calcination at 450 °C to produce fine, black NiO particles. The produced nanoparticles were methodically studied by FTIR and UV–visible spectroscopy to examine their structural, chemical, and optical properties.

Methodology

Chemicals and Reagents

All reagents were analytical-grade and used without further purification. These included nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$), ammonium hydroxide, cellulose acetate (CA), acetone (99% purity), copper sulfate ($CuSO_4 \cdot 5H_2O$), silver nitrate ($AgNO_3$), sodium borohydride ($NaBH_4$), and deionized water (resistivity ≥18.2 MΩ·cm).

Synthesis of NiO Nanoparticles via Sol–Gel Method

NiO nanoparticles were synthesized by a sol–gel approach. In one route, agarose polysaccharide was used as a templating agent, and the precursor gel was calcined at 400 °C to obtain NiO nanoparticles of ~3 nm (Alagiri, Ponnusamy, & Muthamizhchelvan, 2012). In another route, nickel nitrate and ammonium hydroxide were used to prepare a gel precursor, which was then calcined at 400–1000 °C, yielding crystalline NiO with average crystallite sizes between 12 and 20 nm (Kayani, Butt, Riaz, & Naseem, 2018).

Preparation of Cellulose Acetate–NiO (CA–NiO) Nanocomposites

Cellulose acetate (4 g) was dissolved in acetone (99% purity) under magnetic stirring

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until a clear solution was obtained. NiO nanoparticles (0.1 g, synthesized as described above) were dispersed in the polymer solution, and the suspension was stirred at room temperature to ensure homogeneity. The mixture was then cast onto glass substrates using a glass rod. After solvent evaporation under ambient conditions, the dried thin films were peeled off and stored for further use (Alamry & Ali, 2020).

Metal Ion Uptake by CA and CA–NiO Films

Aqueous solutions of $CuSO_4$, $AgNO_3$, and a binary mixture of $CuSO_4$ and $AgNO_3$ (2 g each) were prepared in double-distilled water with mechanical stirring until fully dissolved. The prepared CA and CA–NiO films were immersed in these solutions for 5 h to facilitate adsorption of Cu^{2+}, Ag^{+}, or mixed ions (Islam, Mishra, & Patel, 2011).

Formation of Metal Nanoparticles on Films

Following ion uptake, the loaded films were immersed in $NaBH_4$ solution to reduce the adsorbed metal ions. This process yielded Ag^0/CA, Cu^0/CA, Cu^0–Ag^0/CA, Ag^0/CA–NiO, Cu^0/CA–NiO, and Cu^0–Ag^0/CA–NiO nanocomposites. A distinct brown color indicated successful nanoparticle formation (Alamry & Ali, 2020).

Catalytic Reduction of 4-Nitrophenol (4-NP)

The catalytic activity of the nanocomposites was assessed using the reduction of 4-NP to 4-aminophenol (4-AP) in the presence of $NaBH_4$. Typically, aqueous 4-NP (100 mL, 0.15 mmol) and $NaBH_4$ (100 mL, 15 mmol) were mixed in a sealed quartz cuvette. UV–vis spectra (200–800 nm) were recorded at 1-min intervals. The decrease of the 4-NP band at ~400 nm and the appearance of a 4-AP band at 317 nm confirmed the reaction (Kuroda, Ishida, & Haruta, 2009; Yu, Tu, & Xu, 2010).

Results

Fabrication of CA–CuO Nanocomposite Films

A cellulose acetate (CA)–CuO nanocomposite film was successfully fabricated by dissolving 4 g of CA in acetone and incorporating 0.1 g of CuO nanoparticles. The resulting solution was cast onto glass substrates and dried to form uniform composite layers. Pure CA films were prepared under identical conditions without CuO for comparison.

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Figure 1 different solutions of Copper for synthesis of NPs

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Figure2 Temperature used for synthesis of NPs

Metal Ion Adsorption and Reduction

Immersion of CA and CA–CuO films in aqueous solutions of $AgNO_3$, $CuSO_4$, or a mixture of both resulted in distinct color changes, indicating metal ion adsorption. The films turned blue in Cu^{2+} solution, dark red in Ag^{+} solution, and bluish-red in Cu–Ag mixtures. Upon subsequent treatment with $NaBH_4$, rapid color shifts occurred within minutes, corresponding to the reduction of the adsorbed ions into metallic nanoparticles. The transformation produced Cu^0, Ag^0, and Cu–Ag nanoparticles on both CA and CA–CuO supports. Among these, the CA–CuO-based system demonstrated the highest catalytic activity in the reduction of 4-nitrophenol to 4-aminophenol.

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Figure 3 Drying process of NPs

Morphological and Elemental Characterization

Field emission scanning electron microscopy revealed morphological differences between CA, CA–CuO, and the nanoparticle-loaded composites. Energy-dispersive X-ray spectroscopy confirmed the elemental composition of each sample. Pure CA exhibited only carbon and oxygen peaks, while CA–CuO additionally showed a minor copper peak. After metal loading and reduction, distinct signals of silver, copper, or both were observed in the corresponding composites, confirming successful nanoparticle incorporation.

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Figure 4 NPs weighing on digital balance for making solutions

Structural Analysis

X-ray diffraction patterns of pure CA films showed an amorphous hump with no crystalline peaks. The CA–CuO composite displayed a similar pattern with suppressed CuO peaks due to its low concentration. In contrast, the metal-loaded CA–CuO films exhibited distinct reflections corresponding to metallic nanoparticles. A strong peak at $2\theta \approx 45°$ was assigned to the (111) plane of face-centered cubic copper, confirming the formation of Cu^0 nanoparticles. Crystallite size calculations indicated average values of 7.7, 9.94, and 12.4 nm across different samples.

Functional Group Analysis

Fourier-transform infrared spectroscopy of CA films showed characteristic absorption bands at 3480 cm^{-1} (O–H stretching), 2934 cm^{-1} (C–H stretching), 1734 cm^{-1} (C=O stretching), and 1029 cm^{-1} (C–O stretching). The CA–CuO nanocomposite exhibited a similar spectrum with an additional absorption band in the 500–600 cm^{-1} range, corresponding to metal–oxygen vibrations. Metal-loaded CA–CuO composites displayed the same functional group peaks but with slight shifts in O–H, C=O, and C–O bands, indicating interactions between the polymer matrix and the nanoparticles.

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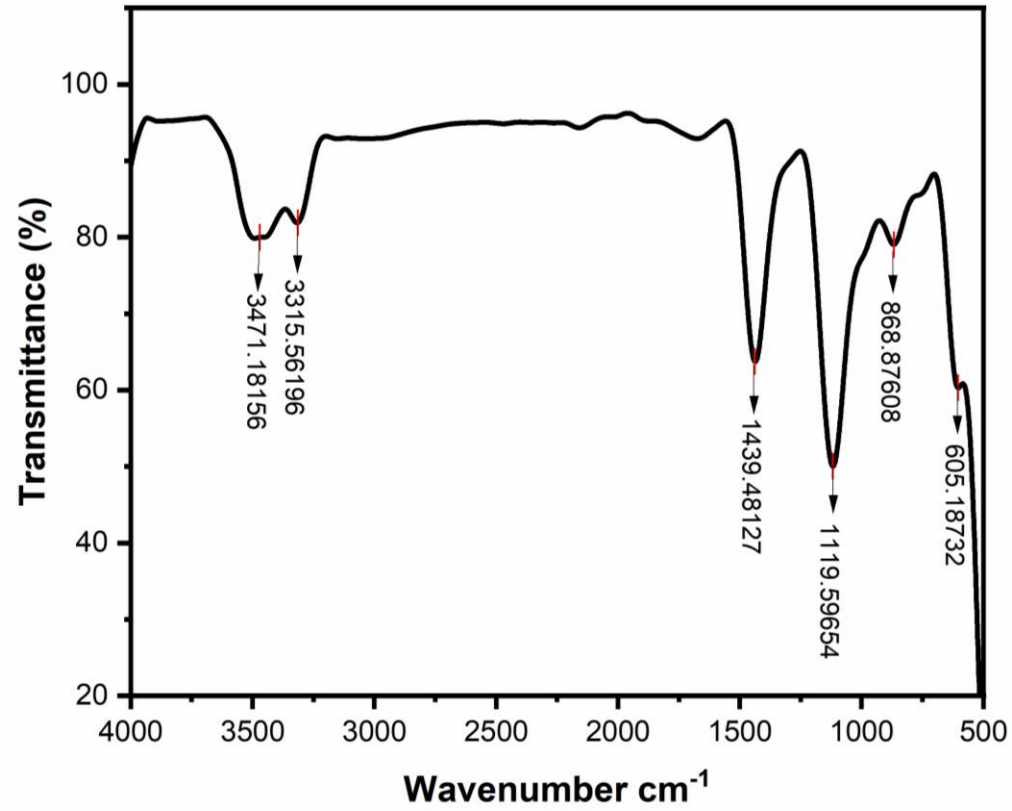


Figure 5 FTIR pattern of Nickel Oxide nanoparticle

Optical Properties of NiO Nanoparticles

UV–visible spectroscopy of NiO nanoparticles showed a distinct absorption peak at 328 nm, corresponding to intrinsic optical transitions. The sharp absorption edge suggested good crystallinity and confirmed their semiconducting nature.

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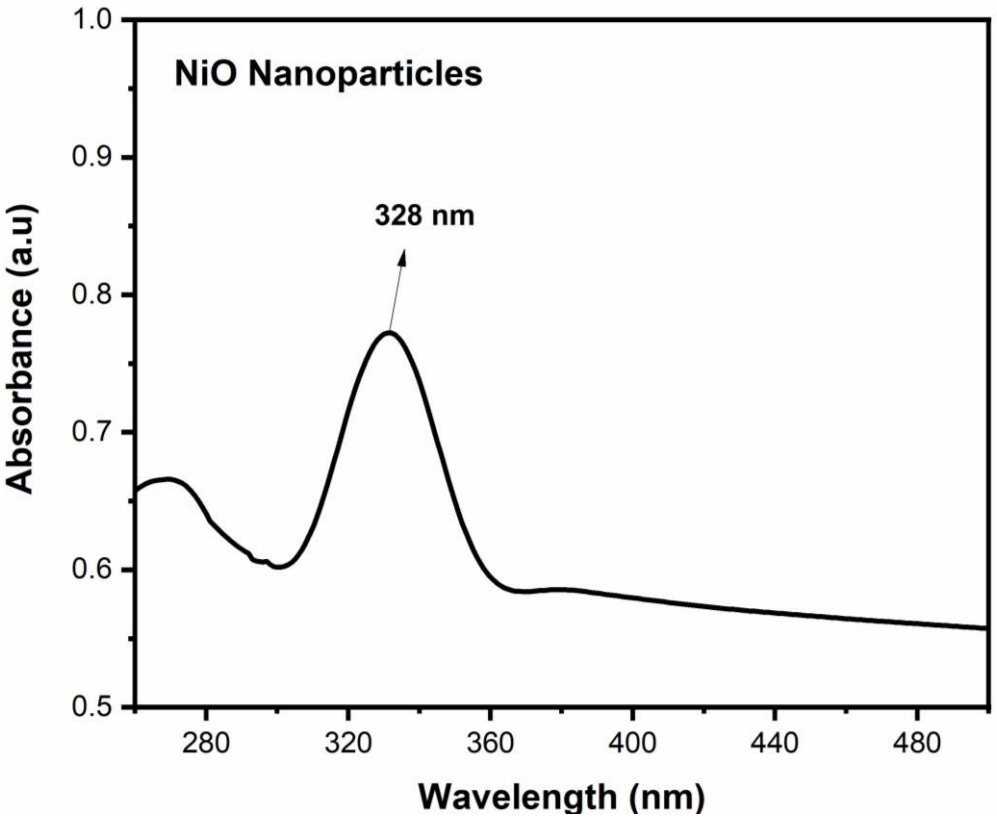


Figure 6 . UV/Visible Spectra of NiO

Vibrational Properties of NiO Nanoparticles

Fourier-transform infrared spectroscopy of NiO nanoparticles revealed a broad absorption band at 3200–3600 cm^{-1}, attributed to hydroxyl stretching vibrations. Peaks in the range of 2800–3000 cm^{-1} corresponded to C–H stretching, while an absorption band at 1650–1750 cm^{-1} indicated the presence of C=O groups. A strong band in the 400–600 cm^{-1} region was assigned to Ni–O stretching vibrations, confirming the formation of nickel oxide. The absence of additional peaks suggested high phase purity of the synthesized nanoparticles.

Discussion

This study illustrates the efficient adsorption and in situ reduction of metal ions on cellulose acetate–copper oxide (CA–CuO) composite sheets. The significant color alteration noted upon immersion in Cu^{2+}, Ag^{+}, and Cu–Ag ion solutions, followed by $NaBH_4$ treatment, substantiates the anticipated redox conversion into metallic Cu^0, Ag^0, and Cu–Ag^0 nanoparticles. This behavior corresponds with previous findings on cellulose acetate-stabilized Cu_2O nanoparticle systems, where analogous chromatic changes indicated metal reduction and nanoparticle production (Benhadria et al., 2025). The many hydroxyl groups in the cellulose acetate matrix promote ion binding and reduction, reflecting mechanisms observed in cellulose-supported metallic catalysts (Kamel & Khattab, 2021).

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The FESEM and EDS data validate nanoparticle deposition without polymer degradation, corroborating findings that cellulose nanocrystals serve as an eco-friendly stabilizing template for CuO nanoparticles, maintaining both dispersion and composite shape (Zhou et al., 2013; Ahmad et al., 2024). XRD results indicate distinct reflections at $2\theta \approx 45°$, corresponding to the Cu^0 (111) plane, with crystallite sizes ranging from around 7 to 12 nm, aligning with standard dimensions of metal nanoparticles that enhance surface-area-to-volume ratios and catalytic performance (Pathak et al., 2024).

FTIR spectra demonstrate shifts in O–H, C=O, and C–O bands, signifying interfacial interactions between the polymer and nanoparticles. These spectrum alterations resemble coordination effects in biopolymer-stabilized catalytic systems, where shifts result from hydrogen bonding or complexation between the polymer and metal centers (Moores & Kaushik, 2016). The UV–Vis absorption at 328 nm highlights the semiconducting properties and crystalline integrity of the NiO nanoparticles, attributes essential for catalysis and photocatalysis, consistent with similar investigations on NiO optical transitions (Gupta et al., 2015).

The elevated purity of NiO, confirmed by FTIR bands (400–600 cm^{-1} for Ni–O stretching) exhibiting few impurity peaks, corresponds with the structural integrity documented for meticulously manufactured nickel oxide nanostructures (Baruah & Dutta, 2009; Ristić et al., 2005). The results demonstrate that the composite system offers a strong and versatile catalytic platform.

The enhanced catalytic activity in the reduction of 4-nitrophenol by CA–CuO-supported nanoparticles is ascribed to the synergistic dual functionality of CuO integrated within CA and the deposited metallic nanoparticles. CuO may function as a catalyst for electron transport or as a repository for Cu^+/Cu^0, similar to the increased activity observed in CuO–NiO binary systems (Ali et al., 2024). The cellulose acetate matrix facilitates dispersal and reduces nanoparticle agglomeration, corroborating earlier research that endorses environmental stability and recyclability (El-Hajjaji et al., 2025; Kaushik & Moores, 2016).

The CA matrix may function as both an adsorption scaffold and an electron conduction medium, facilitating reductive reactions by stabilizing the intermediate M–NP species. This process corresponds with cellulose-mediated green catalysis models, wherein nanocelluloses function as supports, stabilizers, and in situ reductants (Kaushik & Moores, 2016; Benhadria et al., 2025). The detection of M–O shifts in FTIR and distinctive XRD reflections indicates interface interactions that may reduce activation barriers for the catalytic reduction process.

Notwithstanding encouraging outcomes, additional scrutiny—encompassing kinetic analysis (turnover frequency, rate constants), recyclability evaluations, and mechanistic exploration through in situ spectroscopy—will be imperative for thorough assessment. Comparative analyses with various polymer matrices or metallic compositions (e.g., pure cellulose, different metal oxides) could clarify the roles of CA, CuO, and metallic nanoparticles in catalytic efficacy (Kamel & Khattab, 2021; Pathak et al., 2024).

This study demonstrates that cellulose acetate–CuO composites are effective supports for the creation of metal nanoparticles, with potential uses in green catalysis. This system's integration of polymer matrix stabilization, controlled nanostructure creation, and catalytic activity renders it suitable for environmental remediation technologies.

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Conclusion

In order to successfully synthesis NiO nanoparticles, a straightforward and cost-effective sol–gel approach was utilized. This process resulted in the production of high-purity materials that exhibited semiconducting behavior and obvious Ni–O links. Applications in optoelectronics, catalysis, and environmental remediation are all areas that could benefit greatly from the nanoparticles' significant potential, which is highlighted by their optical and structural features. It is a potential technique for the industrial manufacture of nanomaterials based on nickel oxide (NiO) due to the fact that the sol–gel process is both scalable and inexpensive. In the future, research should concentrate on optimizing synthesis parameters, such as calcination settings and precursor composition, in order to further customize particle size, shape, and electrical characteristics for particular technological applications.

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