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PROBING STRUCTURAL AND FUNCTIONAL GROUP FEATURES OF NICKEL OXIDE TOWARD IMPROVED DRUG LOADING EFFICIENCY

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Article Info

Abstract
Nanotechnology has emerged as a transformative field in biomedical science, providing innovative approaches for disease detection, diagnosis, and therapy through the design of functional nanomaterials that interact precisely with biological systems. Among the various nanomaterials, nickel oxide (NiO) nanoparticles have attracted considerable attention due to their excellent chemical stability, affordability, and versatile physicochemical properties, which make them promising candidates for applications ranging from drug delivery to imaging and therapeutic platforms. In this study, NiO nanoparticles were synthesized using a simple, reproducible, and cost-effective chemical co-precipitation method, chosen for its scalability and suitability for large-scale production. The prepared NiO nanoparticles were subjected to Fourier Transform Infrared Spectroscopy (FTIR) to investigate their structural features and identify functional groups present on their surface, as these characteristics play a decisive role in drug–nanoparticle interactions. The outcome of this study provides a foundation for the rational design of NiO-based nanocarriers and highlights the importance of further investigations into particle size control, surface functionalization strategies, and biocompatibility assessments to optimize their performance for advanced drug delivery systems and clinical applications.

Keywords:
Nickel oxide, Fourier Transform Infrared Spectroscopy, Co-precipitation, Nanocarriers, Drug loading

INTRODUCTION

Metal oxide nanoparticles (MO-NPs) have emerged as highly promising candidates for use in biomedical applications, particularly in drug delivery systems, owing to their distinct physicochemical and biological properties [1-3]. Nanomaterials such as cerium oxide, zinc oxide, iron oxide, and titanium oxide exhibit several advantageous properties, including high surface reactivity, adjustable structural characteristics, a large surface-to-volume ratio, and improved chemical and thermal stability [4]. Cobalt oxide is composed of multiple valence states, containing both Co^{2+} and Co^{3+} ions. It crystallizes in a cubic structure, where Co^{3+} ions occupy octahedral sites and Co^{2+} ions are located in tetrahedral sites [5–11].

Several techniques have been explored for the fabrication of metal oxide nanoparticles, such as thermal decomposition [12], pyrolysis [13], microemulsion techniques [14], ultrasonic irradiation [15], sol–gel [16,17], hydrothermal synthesis [18] and microwave-assisted methods [19]. For instance, Irudaya et al. [20] synthesized pure and Li-doped NiO nanoparticles via sol–gel method, which resulted in cubic phase nanoparticles exhibiting a blue shift and potential applications in catalysis and gas sensing. Similarly, Kalam et al. [21] produced NiO nanoparticles using the thermal decomposition of nickel linoleate and studied their optical characteristics. Despite their effectiveness, many of these techniques demand high temperatures, pressures, sophisticated equipment, and costly precursors, limiting their scalability.

Biologically inspired synthesis approaches help prevent nanoparticle agglomeration while enhancing stability and surface activity, making them particularly suitable for biomedical applications. Green-synthesized NiO nanoparticles have demonstrated significant antibacterial, antifungal, antioxidant, anticancer, anti-inflammatory, and antiparasitic effects, while also showing relative safety at controlled concentrations. Their biological functions are often attributed to the generation of reactive oxygen species (ROS) and the release of Ni^{2+} ions, which can disrupt microbial structures or induce apoptosis in cancer cells [8]. Despite these advances, most prior research has concentrated primarily on synthetic methods, physicochemical characterization, or broad-spectrum biological effects, while relatively little attention has been given to probing the structural and surface functional group features of NiO nanoparticles that directly influence drug–nanoparticle interactions. Functional groups such as hydroxyl, carboxyl, and carbonyl play an essential role in drug binding, encapsulation, and controlled release behavior, making their investigation crucial for developing effective nanocarrier systems [9]. To address this research gap, the present thesis focuses on synthesizing NiO nanoparticles via a chemical co-precipitation method, characterizing their structural and surface features through Fourier-transform infrared spectroscopy (FTIR), and correlating these attributes with their drug loading efficiency. By establishing a direct link between material properties and biomedical performance, this work aims to contribute toward the rational design of NiO-based nanocarriers for advanced therapeutic and biomedical applications [10].

Nanomaterials can be synthesized through chemical or physical methods. chemical approach not only reduces toxicity but also introduces biologically active functional groups, such as hydroxyl, carbonyl, and amino groups, onto the nanoparticle surface, which can significantly enhance drug adsorption and interaction with biomolecules. Moreover, green-synthesized NiO nanoparticles demonstrate higher biocompatibility compared to chemically synthesized ones, making them highly suitable for biomedical applications [11]. Several studies have reported that these nanoparticles exhibit antimicrobial, anticancer,

and antioxidant activities, largely due to their surface chemistry and ability to generate reactive oxygen species (ROS) in controlled ways. Importantly, the environmentally sustainable nature of green synthesis aligns with the increasing demand for safe and scalable nanomaterial production, particularly in pharmaceutical and therapeutic contexts where toxicity and patient safety are of critical importance. Thus, green synthesis not only addresses the limitations of traditional physicochemical routes but also provides an added functional advantage by integrating surface groups favorable for drug binding and controlled release [4-7].

Synthesis-Protocol

Chemicals: (1) Nickel nitrate hexahydrate [$Ni(NO_3)_2 \cdot 6H_2O$], (2) Qualigens [Co. Ltd.] and (3) sodium hydroxide pellets (Merck). All chemicals are of analytical grade with a purity of ≥99%.

Methodology: Two separate aqueous solutions were made: one by dissolving $Ni(NO_3)_2 \cdot 6H_2O$ in deionized water, and the other by dissolving NaOH pellets in deionized water. The NaOH solution was then added dropwise into the nickel nitrate solution under continuous stirring at 50 °C for half hour. Constant stirring for an additional 1h to ensure homogeneous mixing, leading to the formation of a light green precipitate. The precipitates were repeatedly washed with deionized water to eliminate any residual ions or impurities, followed by filtration using Whatman filter paper. The obtained powder was desiccated at 150 °C for 11 h under ambient conditions, yielding a green solid mass. This dried material was finely ground in mortar and pestle for 30 min. Subsequently, the powder was calcined at 300 °C for 2 h, during which the color changed from green to black, confirming the formation of NiO. Finally, the calcined powder was again ground thoroughly for 45 min and stored for more characterization.

The flowchart for whole followed process is explained shown in Fig 1.

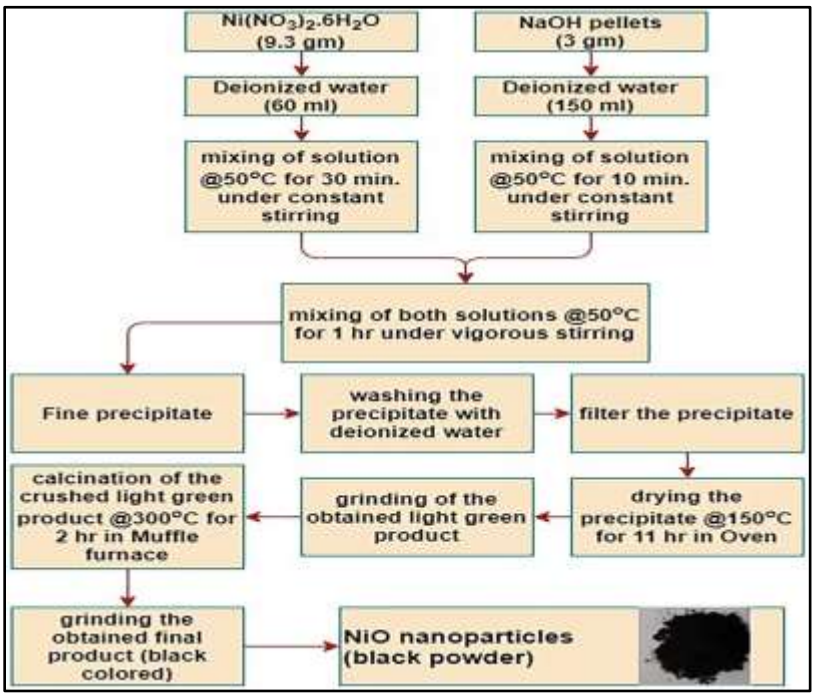

Fig. 1: Flowchart for the synthesis of nickel oxide NPs

Results and Discussion

Fourier Transform Infrared Spectroscopy (FTIR) is an advanced spectroscopic technique used to analyze the vibrational behavior of chemical bonds in a sample. It works by exposing the material to infrared radiation and recording the absorbance or transmittance at different frequencies. Since each functional group absorbs IR radiation at a characteristic frequency, the resulting spectrum acts as a unique "molecular fingerprint," enabling researchers to identify structural features, functional groups, and possible impurities. FTIR primarily provides detailed information about the composition and nature of chemical bonds within a material. When infrared radiation is directed onto a sample, the molecules absorb energy and undergo vibrational excitations. These vibrations occur when the frequency of the incident IR radiation matches the natural vibrational frequency of the molecular bonds. By adjusting the frequency of the radiation, different vibrational modes can be probed, allowing identification of specific functional groups in the material. The role of Fourier transformation in this process is mathematical. The instrument initially records an interferogram, which represents the raw data in the time domain. Fourier transformation is then applied to convert this data into the frequency domain, producing a spectrum of absorbance or transmittance versus wavenumber. This spectrum is easier to interpret, as each peak corresponds to a characteristic bond vibration.

Fourier Transform Infrared (FTIR) spectroscopy was used to determine the surface functional sets and bonding characteristics of the synthesized NiO nanoparticles in the wavenumber region of 600–4000 cm^{-1}, as depicted in Fig. 2. The absorption peak observed near 620 cm^{-1} is a characteristic vibrational mode of Ni–O stretching, which provides clear evidence for the formation of nickel oxide nanoparticles. The presence of this band is a strong confirmation of the crystalline NiO phase since it corresponds to metal–oxygen lattice vibrations typically reported in transition metal oxides. In addition to the Ni–O vibration, a broad absorption band was detected in the range of 3400–3800 cm^{-1}. This band is attributed to O–H stretching vibrations, which arise due to hydroxyl groups. The broad nature of this FTIR band indicates the presence of hydrogen-bonded water molecules physically adsorbed on the surfaces of the NiO nanoparticles. Such surface hydroxylation is a characteristic feature of nanostructured oxides, arising from their high surface-to-volume ratio, which increases the number of active sites available for moisture adsorption. This property not only affects the hydrophilicity of the nanoparticles but can also influence their catalytic, adsorption, and drug-loading behavior.

A distinct absorption band observed near 1639 cm^{-1} corresponds to H–O–H bending vibrations, providing further evidence of molecularly adsorbed water on the nanoparticle surface [23]. The persistence of this water signal even after calcination suggests that water molecules are strongly bound to the NiO surface, potentially through hydrogen bonding with surface hydroxyl groups.

In addition, weak absorption bands appearing around 1366 cm^{-1} and 1742 cm^{-1} are attributed to C=O stretching vibrations, indicative of carbonyl groups on the nanoparticle surfaces. These carbonyl species are typically formed through the interaction of atmospheric CO_2 with surface hydroxyl groups, resulting in carbonate-like bonds. The presence of these groups highlights the strong surface reactivity of NiO nanoparticles, particularly their ability to interact with gaseous molecules. Such surface chemistry is crucial for applications in catalysis, gas sensing, and environmental remediation, as it enhances the

adsorption and activation of reactant molecules. Overall, the FTIR analysis confirms that the NiO nanoparticles possess a highly active surface with adsorbed water and carbonate species, which can significantly influence their functional performance in various applications [22].

The combined presence of hydroxyl and carbonyl groups in the calcined sample highlights the strong adsorption tendency of NiO nanoparticles towards water vapor and carbon dioxide from the surrounding atmosphere. This adsorption behavior is important since surface functionalization plays a critical role in determining the catalytic, sensing, and electrochemical properties of NiO. Similar findings regarding the adsorption of H_2O and CO_2 on NiO surfaces have also been reported in previous studies [24, 25], confirming that the FTIR results obtained in this work are consistent with earlier literature.

Table 1: Functional groups assigned to corresponding frequencies in FTIR

Sr.	Functional group assigned	IR frequencies (cm^{-1})
1.	υ_s (Ni–O)	620
2.	υ (C = O)	1742
3.	δ_s (H–O-H)	1639
4.	υ_{as} (O–H)	3840
5.	υ_s (C-O)	1366
6.	υ_s (O–H)	3454

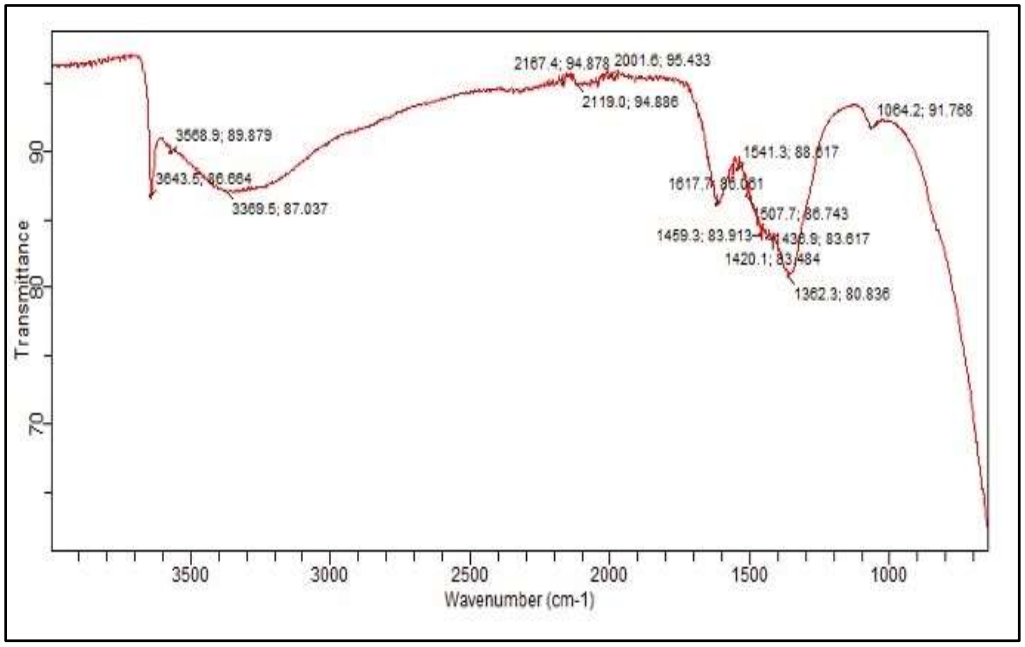

Fig. 2: FTIR result of the prepared nickel oxide NPs

Conclusion

In this research, nickel oxide (NiO) nanoparticles were synthesized through a chemical co-precipitation route, which proved to be an effective, low-cost, and scalable method for producing nanostructures with well-defined properties. The approach offered control over crystallinity, particle size, and morphology by regulating synthesis conditions such as precursor concentration, solution alkalinity, and calcination temperature. Structural analysis confirmed the formation of crystalline NiO, while Fourier Transform Infrared Spectroscopy (FTIR) detected characteristic Ni–O vibrations along with hydroxyl and carbonyl groups on the nanoparticle surfaces. These surface functional groups are particularly important for biomedical use, as they govern the interaction of NiO nanoparticles with therapeutic molecules. However, despite these advantages, challenges related to cytotoxicity, biodistribution, and long-term biological safety remain unresolved and must be addressed before clinical translation. Future investigations should therefore aim to optimize synthesis parameters for greater control over nanoparticle uniformity, explore advanced surface modification techniques such as polymer encapsulation, ligand grafting, and biomolecule conjugation to enhance biocompatibility, and incorporate computational simulations to better predict drug–nanoparticle interactions at the molecular level. Further in vitro and in vivo studies are also needed to evaluate drug release profiles, cellular uptake pathways, and pharmacological responses in real biological systems. Overall, this work demonstrates that the structural and surface chemistry features of NiO nanoparticles directly influence their drug-loading efficiency and therapeutic potential, laying the groundwork for their development as versatile nanocarriers in next-generation drug delivery and nanomedicine platforms.

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