

NICKEL OXIDE AS A POTENTIAL ANTIMICROBIAL AGENT: SYNTHESIS AND CHARACTERIZATION

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Abstract

Nickel oxide (NiO) nanoparticles have a natural bactericidal activity that can be further improved by smart doping with other metallic elements. In this work, NiO nanoparticles were prepared by sol-gel method at different concentration to understand their synergistic antimicrobial activity. The successful incorporation of dopants into the NiO lattice and their successful synthesis was confirmed by Fourier Transform Infrared (FTIR) spectroscopy. NiO nanoparticles also showed better performance, thus they hold the promising potential for their applications in the field of medical coatings, disinfectants and as new antibacterial agents used for the fight against drug-resistant microbes.

Keywords:

Nickel Oxide (NiO), Nanoparticles, Antimicrobial Activity, Sol-Gel Method

INTRODUCTION

Nanotechnology is the science, engineering and application of materials and devices at the nanometer scale (1100 nanometers). Matter must be processed to the atomic or molecular scale in order to form structures of special properties. The field is multidisciplinary and all the areas namely physics, chemistry, biology, medicine, engineering and materials science, are covered. The materials that exhibit one of their dimensions 1 to 100 nanometers (nm) in size are called nanoparticles. The size of nanoparticles is too small which makes their properties different to those of bulk substances. These individual characteristics can be attributed to, amongst others, high surface-to-volume proportion, quantum estimations and more surface reactivity. Nanoparticles may exist in naturally occurring and synthetically produced substances and they have wide applications in many areas, especially in the fields of healthcare, electronics, renewable energy, and the interesting field of environmental science (1).

Nanotechnology is simply the manipulation of matter in the atomic and molecular scale (typically between one to one hundred nanometers). It has grown to be a futuristic concept into a major driver of innovation in most of the scientific and industrial spheres. Because it also allows material and device engineering with substantially novel properties generated precisely because of their small dimensions and large surface-area to volume ratio, unprecedented capabilities are made possible. Medical equipments, energy, environmental sciences, electronics, and materials engineering: all very important industries that are dynamically searching for this transformative potential (2).

In medical discipline, nanotechnology gets a innovative approach to analysis and treatment commonly known as nanomedicine. Possibly, one of its most important uses is in battered drug delivery using plotted nanoparticles. These carriers have the potential to enhance efficacy significantly by targeting therapeutic agents to sick cells or tissues and minimizing side effects to the systemic side effects associated with conventional treatments of cancer such as chemotherapy (3). Instantaneously nanotechnology is having an outstanding effect on diagnostics with the increase of highly complex nano sensors (4)(5). Pollution control plans also call for nanomaterials. Due to their reactivity and adsorbing power, they are effective agents to absorb and break down the air and water pollutants and thus, leading to the development of cleaner ecosystems; the pollutants including heavy metals, organic compounds and greenhouse gases (6).

Finally, materials science and nanotechnology affect immensely. Due to invention and development of nanomaterials such as graphene, carbon nanotubes, structural materials and composites based on great properties have been developed. They are more impressive than well-studied conventional materials such as steel or aluminum by the outstanding level of lightness and electrical and thermal conductivity as well as a high tensile strength. This allows the development of materials that are tougher, lighter and sturdier to be used in consumer products, structures, cars, and aero special sectors (7).

The applications of nanotechnology are now being involved in virtually every area of modern science and technology, and they are growing steadily. Antimicrobial activities discuss the capability of a substance (chemical, nanoparticle, or natural compound) to **prevent growth or destroy microorganisms**, including viruses, bacteria, parasites and fungi. These activities are critical in medication, farming, and food preservation to fight infections and decomposition.

Types of Antimicrobial Agents

Category	Examples	Target Microbes
Antibiotics	Penicillin, Ciprofloxacin	Bacteria
Antifungals	Fluconazole, Amphotericin B	Fungi (e.g., Candida)
Antivirals	Oseltamivir (Tamiflu), Remdesivir	Influenza, SARS-CoV-2
Metal Nanoparticles	AgNPs, CuNPs, NiCl ₂	Broad-spectrum

Nickel chloride has become an antimicrobial agent because of its despite its proven ability to kill wide spectrum of harmful microorganisms. The antimicrobial effects of the compound are achieved in many ways. The main effect of Ni²⁺ ion activity with bacterial cell membranes is the high degree of permeability and distraction of the structure that ended in increasing cell death (8). Moreover, nickel chloride causes microbial oxidative stress by the formation of reactive oxygen species or ROS that affect the cellular components (protein and DNA). In addition to out-competing on rate-limiting metalloenzymes critical in pathways, the antimicrobial activity is further enhanced by active site binding that prevents essential metabolic processes(9). Due to these synergistic effects, nickel chloride has the specific usefulness of antidrug-resistant pathogens, including multidrug-resistant strains of *Escherichia coli*, *Pseudomonas aeruginosa* biofilms, and methicillin-resistant *Staphylococcus aureus* (MRSA).

Antimicrobial properties of nickel chloride have been explored in many clinical applications in the recent past. Polymers with NiCl₂ showed exceptional accomplishments in clinics with a lower 78 percent infection rate of catheters and sustained efficacy in 30 days of active use (Adwin Jose et al., 2024)(8).

Experimental Section: Sol-Gel Method

The sol-gel method is a versatile technique which is fabricating advanced inorganic and hybrid materials which were pioneered in the mid of 19th century. This process gained popularity among scientists as early as 1980 for preparing glasses, high purity ceramics and nanocomposites without sintering. This process consists of the transformation of the colloidal suspension (sol) to an interconnected net 3D structure (gel) through hydrolysis and condensation reactions. Its mechanism is based on the polymerization of inorganic salts or metal alkoxides forming metal-oxo (M-O-M) bonds that result in porous solid networks. This method allows some atomic-scale control over alkoxides ore, composition and functionality, which makes it essential for materials engineering and modern nanotechnology(10).

The importance of sol-gel method lies in its ability to design nanomaterials in mild conditions (25-100 Celsius degrees). Unlike conventional processes in which high temperature is required, this method allows us to maintain thermally sensitive phases and allows us to integrate organic polymers. It leads to molecular homogeneity and makes sure that dopants have been dispersed uniformly such as Cu and Ag in NiO for increased functional characteristics. Porosity and precise control over surface area substitute its applications in biomedicine and energy storage (11).

Synthesis and Characterization of Nickel-Based Nanomaterials: Experimental Methodology

Materials

The nickel-based nanomaterials was prepared by a sol-gel method using nickel chloride hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$), sodium hydroxide (NaOH , pellets), and citric acid monohydrate ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$) as major precursors. Deionized water ($\text{DI H}_2\text{O}$) used as the universal solvent in this procedure. The experimental workflow started with the careful preparation of precursor solutions. Especially, a 0.25 M nickel chloride solution was formulated by dissolving 5.9 g of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ in 100 mL of deionized water under certain conditions. At the same time, a 1 M citric acid solution was prepared by dissolving 1.9 mL of the compound in 10 mL deionized water, and a 1 M sodium hydroxide solution was obtained by dissolving 4 g of NaOH pellets in 100 mL deionized water (12).

Nanomaterial Synthesis Methodology

The precipitation reaction was initiated by combining 50 mL of the 0.25 M NiCl_2 solution with 25 mL of the 1 M citric acid solution within a glass beaker. This mixture was vigorously agitated using a magnetic stirrer for a duration of 10 minutes at room temperature. During this stage, the formation of a soluble nickel-citrate complex was evidenced by the development of a clear green solution, confirming the chelation of Ni^{2+} ions by citrate anions. This process is critical for modulating further particle nucleation and growth dynamics. Ethylene glycol was added to the solution shortly after preparing nickel-citrate solution. This catalyzed a polymeric templating agent to undergo Poly esterification with citrate in heating conditions to provide a porous metal-organic network that regulates the morphology-controlling nanoparticles by inhibiting agglomeration. The mixture was observed to have raised viscosity, which attested that the polymer had formed a gel before the precipitation occurred (13).

After the formation of complex, the 1.0 M NaOH solution was introduced dropwise at a controlled rate into the stirring nickel-citrate mixture. The addition of NaOH solution was performed with continuous stirring maintained to assure homogenous distribution and forbid localized concentration gradients. This process was crucial to control the pH increase and supersaturation level. An immediate formation of a fine green precipitate was observed upon the addition of NaOH . This indicated the formation of nickel hydroxide. The reaction mixture was further stirred for 30 minutes after the completion of NaOH addition. This ensured the precipitation reaction was completed and initial particle was matured (14).

After precipitation, the reaction mixture was subjected to thermal treatment phase in order to promote particle growth, potential dehydration and phase transformation. The beaker containing the suspension was put on pre-heated heating mantle at a temperature range of 250–300°C. A target operational temperature of $95 \pm 5^\circ\text{C}$ was managed by a thermometer. The suspension was continuously stirred at a reduced rate of 300 rpm for a period of 2 hours under these elevated temperature conditions. This heating facilitated Ostwald ripening, hence increasing crystallite size and uniformity. Furthermore, the increased temperature initiated the dehydration of the nickel hydroxide precursor towards the formation of nickel oxide, evident through a color change. Similarly, the citrate species acted as a structural directing agent, and it yielded porous nanostructures. After the thermal aging process, the suspension was carefully

removed from the heating mantle and put in oven for 12 hours. After that it was allowed to cool naturally to room temperature (12). Some steps performed during synthesis are listed in Figure 1.



Figure 1: Experimentation protocol steps

Results and discussion

FTIR spectrum of pure nickel oxide (NiO), the spectrum shows in Figure 2 as material with a relatively simple infrared absorption spectrum (several main absorption peaks and a large amount of transparency in other regions). Most prominent transmittance dips (absorption) are seen at around 1639 cm^{-1} and 994 cm^{-1} . The intense wide band at 1639 cm^{-1} is usually assigned to the H-O-H bending mode of adsorbed water molecules, which is known to be ubiquitous in the amorphous samples of metal oxides exposed to atmosphere. The very intense and sharp peak around 994 cm^{-1} is a fundamental line of NiO, due to the Ni-O stretching vibration in the rock salts crystal lattice. The small peaks at 1383 cm^{-1} and 2358 cm^{-1} were likely due to the atmospheric carbon dioxide (CO_2) interference or to the minor carbonate species, while those in the 3600s cm^{-1} range were characteristic to the O-H stretching vibrations of surface-adsorbed water or hydroxyl groups.

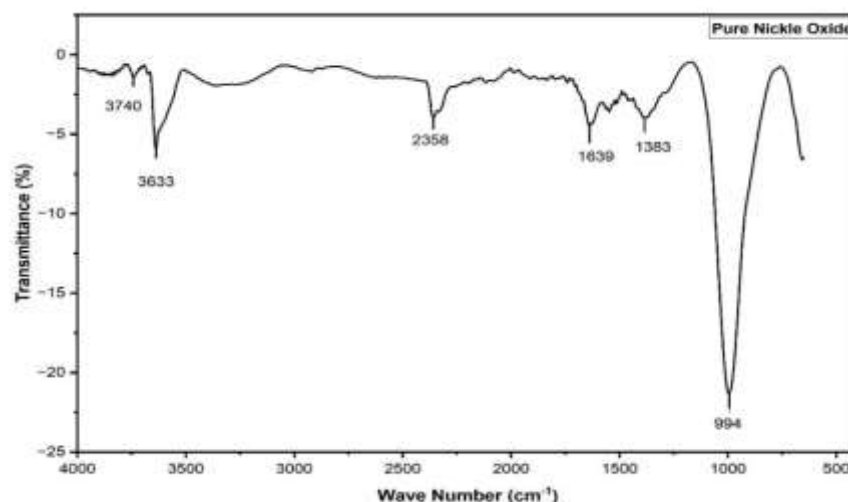


Figure 1: FTIR spectrum of pure nickel oxide

CONCLUSION

The FTIR spectrum of pure nickel oxide shows a material that has a relatively simple chemical structure with all the fundamental vibrational modes of the Ni-O lattice dominating the spectrum. The prominent absorption bands of the metal oxide (Ni-O) stretching and bending vibrations, namely, the strong and broad absorption band centered around $\sim 994\text{ cm}^{-1}$ and the sharper one at $\sim 1383\text{ cm}^{-1}$, respectively, confirm the synthesis of NiO.

In terms of its potential as an antimicrobial, this FTIR data suggests that the mechanism is mainly intrinsic and physical rather than organic/chemical. No strong adsorption bands, such as O-H, N-H, or C=O stretching above 3000 cm^{-1} , and C-H stretching around 2900 cm^{-1} , suggest the absence of organic surface modifiers/capping agents. Therefore, the natural properties of this particular NiO sample can be regarded as the main reason for its high antimicrobial activity:

1. **Direct Interaction:** The antimicrobial action is probably due to the release of Ni^{2+} ion which disrupts microbial cell membranes and produces reactive oxygen species (ROS), causing oxidative stress and cell death.
2. **Physical Disruption:** The nanoparticles can interact directly with and damage the cell walls of micro-organisms.

Overall, the FTIR analysis affirms the purity and inorganic nature of the nickel oxide. This justifies our conclusion that its antimicrobial action is an intrinsic property of the nickel oxide nanoparticles themselves and is not caused by surface-adsorbed organic bioactive compounds. Thus, it shows promise as a pure inorganic agent for antimicrobial use.

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