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ENVIRONMENTAL FRIENDLY SYNTHESIS OF NICKEL NANOPARTICLES AND THEIR BIOMEDICAL APPLICATIONS - A REVIEW

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ABSTRACT
Over the previous decade, nanotechnology has revolutionized the engineering of operating systems at the nanoscale level, most notably in the form of nanoparticles (NPs). Recently nickel oxide nanoparticles (NiO NPs) have been more considered due to their excellent magnetic properties, high surface area, semiconducting nature, complexing tendencies, and reduction capacity. It is used in many fields as catalysts, sensors, and memory storage devices. In addition, it is potentially used for magnetically controlled drug delivery, hypothermia treatment of cancer cells, and magnetic resonance imaging. This review mainly established the synthesis of NiO NPs through the biogenic synthesis process (such as plant-mediated NPs synthesis and some of the microorganisms used to prepare NPS), characterizations, and their potential biomedical applications.

KEYWORDS: Biomedical applications, Nanoparticles, Nickel oxide, Synthesis.

1. INTRODUCTION
Nanotechnology has revolutionized the engineering of operating systems at the nanoscale level over the previous decade, most notably in the form of NPs.[1] Nanoparticles (NPs) are atom clusters with unique optical, magnetic, catalytic, and electrical characteristics. Therefore, they might come in handy in numerous scenarios.[2] Due to their more remarkable magnetic properties and possible applications in multiple disciplines, there has been a spike in interest in synthesizing magnetic NPs of iron, cobalt, and nickel (Ni) in recent years.[3] Ni NPs are divided into two categories: metal and oxide.[4] Novel ink for nanotube-field modulated gratings and optical switches, direct immobilization of biomolecules via magnetic force of Ni NPs. Adsorption of yellow dyes has gotten a lot of attention because of their unusual magnetic, chemical, and physical properties and their potential use in many technology domains like catalysis, battery manufacture, and so on.[5]

Nano-Nickel oxide is also a semiconductor with outstanding chemical stability and electron transport capacity (band gap: 3.6 to 4.9 e V).[6] As a result, these NPs are used in various applications, such as electronics, energy appliances, biomedicine, sensors, waste-water treatment, and numerous biological syntheses based on reduction, hydrogenation, alkylation, etc.[7] Furthermore, they deliver magnetically regulated medications in medicine, perform magnetic resonance imaging, and use hyperthermia to treat tumor cells.[8]

Some of the physical and chemical processes used to generate NPs include co-precipitation, sol-gel, micro-emulsion, hydrothermal reaction, electrospray synthesis, and laser ablation. Although these technologies can make well-defined pure nanoparticles, they are not environmentally friendly and have limited productivity, cytotoxicity, antioxidant capacity, and antibacterial activity.[9]

Natural systems, including bacteria, fungi, yeast, and plants, and naturally happening tiny molecules like vitamins, proteins, peptides, and reducing sugars, are used to create NPs. This work provides a brief overview of the numerous green manufacturing and characterization methods for NiO NPs and their biomedical applications.[10] The figure 1 diagrammatically represents the NiO NPs synthesis, characterization and their applications in biomedicine.

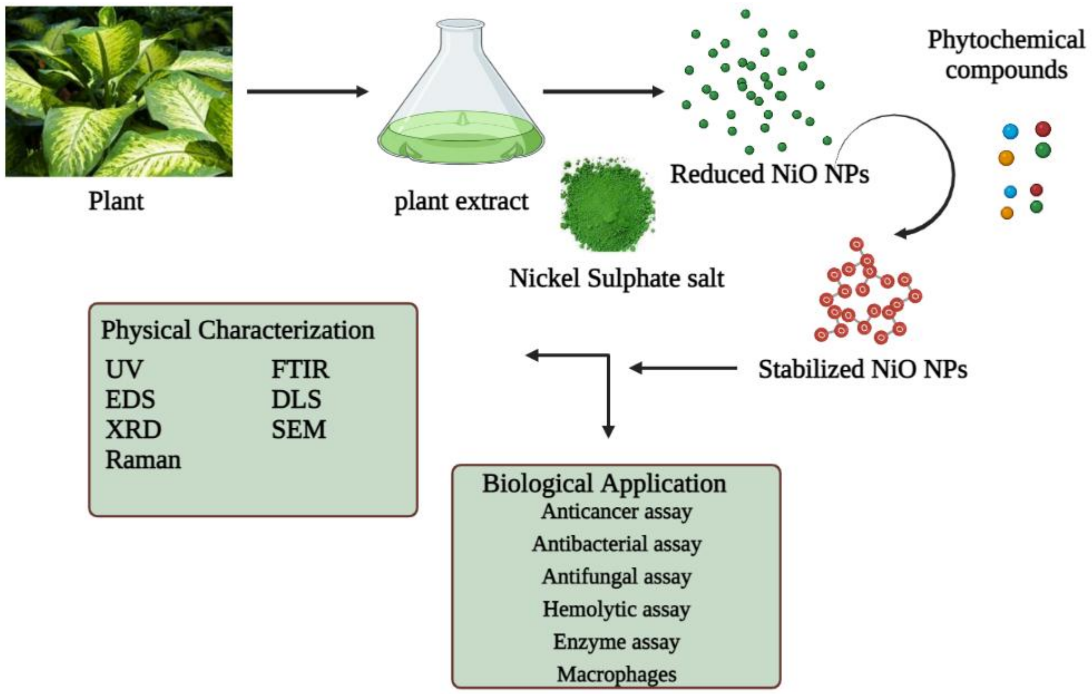

Fig 1: NiO NPs synthesis, characterization, and their applications in biomedicine.

2. Significance of Ni NPs

Electronics, magnetism, energy technology, and biology could benefit from Ni NPs.[11] They are used to catalyze a variety of organic reactions such as chemoselective oxidative coupling of thiols, reduction of aldehydes and ketones, hydrogenation of olefins, synthesis of stilbenes from alcohol over Wittig-type olefination, and -alkylation of methyl ketone due to their high reactivity, operational simplicity, and environmentally friendly things.[12] Inorganic reactions, such as ammonia breakdown, can also be catalyzed. Carbon nanotubes (CNTs) are one of its most recent applications.[13]

Due to its antibacterial and anti-inflammatory characteristics, it acts as an adsorbent for toxic dyes and inorganic pollutants in the environment, contributing to environmental purity.[14] Hence, they are used in biomedicine.[15]

The morphological alterations in these cells following exposure to Ni NPs show cytotoxicity towards malignant cells.[16] Ni NPs encapsulated with biomolecules like glucose has high biocompatibility and were used as biosensors and heat non-mediators to treat malignant hyperthermia.[17]

3. Synthetic methods for Ni NPs

Top-down and bottom-up synthesis methods have historically been used to produce NiO NPs. The nanoscale compound is made by breaking down massive materials through the process of top-down synthesis approaches. Mechanical milling, laser ablation, nanolithography, thermal breakdown, and sputtering are top-down nanoparticle manufacturing methods.[18] MOs and metallic salts are widely utilized as reaction precursors in bottom-up techniques. The suitable solvent and reducing agent convert these salts or oxides into metallic NPs. By varying the precursor and reducing agent concentrations, pH, temperature, heating time, and the type and choice of the stabilizing agent, these methods may alter the dispersal mode, shape, and dimensions of NPs.[19] Several bottom-up methods for creating Nickel Nanosized materials have been published. These procedures include spatting, solvent-gel, gas evaporation, co-precipitation approaches, and plant-mediated synthesis. In non-aqueous and aqueous conditions, chemical techniques, sonication, radiolysis, chemical reduction, and freeze-drying methods have all been employed to produce Ni NPs.[20]

4. Green synthesis of (NiO)

Various green syntheses have synthesized NiO, and some of them are discussed below.

4.1 Synthesis of NiO NPs using plants

In the manufacture of Nickel Oxide Nanocrystals utilizing Nickel sulphate as a precursor, rambutan peel waste was a capping and stabilizing element.[21] *Agathosma betulina* natural extract was used to make nano nickel oxide.[22] Some research has used microbes such as bacteria, yeast, and fungus as reducing and stabilizing agents in the manufacture of Nickel Oxide NPs.[23] Dead biomass from filamentous fungi was employed, while dead biomass from *Rhizopus nigricans* was used.[24] Micro bacterium sp. MRS-1 was used to make nickel oxide microparticles.[25]

All of the particles generated with these bio means have been biocompatible because the capping, reducing, and stabilizing agents are sugars and flavonoids, non-cytotoxic biomaterials.

The available flora contains plant extracts containing compounds that reduce capping agents in stabilizing Nickel and NiO NPs. Plant materials with homogeneous micro-holes may act as templates or substrates. They may aid in creating the exact size NPs of nickel/nickel oxide, as explored concerning the preparation of Nano Ni by Hibiscus flower petals as a bio pattern.[26]

Ni/nickel oxide (isotropic/ anisotropic), by carefully tweaking the synthesis conditions, NPs of specific form and size can be produced, enhancing surface potentials that can be used to bind toxins to the surface. The microbial-mediated generation of nano nickel and nickel oxide is also understudied, and this is an essential topic for research. Surface morphology investigations will be carried out to understand further the mechanics of nickel/nickel oxide nanoparticle formation.

Because of its simplicity, cost-effectiveness, speed, and environmental friendliness, researchers have been lured to manufacture Ni NPs using plants in recent years.[27] Biosynthesis is a one-step approach for creating stable NPs with various morphologies. Compared to microorganism-based NPs biosynthesis, the production rate is significantly faster. Secondary metabolites like terpenoids, flavones, pyrones, aldehydes, amides, and carboxylic acids were discovered to convert metal salts into equivalent NPs using infrared spectroscopy.[28] So far, just a few research papers have been published on plants to generate Ni NPs. NiO NPs are made from plant parts such as leaves and roots.

4.2 Leaf extracts as a reducing agent

It's been proven that *Ocimum sanctum* aqueous leaf extract can be used to produce Ni NPs.[29] They discovered that green nanoparticle production leads to more development in pharmaceutical and biological applications due to its economical and environmentally friendly than chemical and physical techniques. The wet-chemical production of spherical Ni NPs on indium tin oxide surfaces was also demonstrated.[30] According to the researchers, this is the first time that Ni NPs have been linked in situ to an indium tin oxide substrate. The production of Ni NPs using both green and chemical approaches has been disclosed in a publication.[31] According to the researchers, green-synthesized Ni NPs had a smaller and better monodispersity than chemically generated Ni NPs. *Azadirachta* and *Psidium guajava* leaves have been shown to produce Ni nanoparticles.[32] Nickel's mesh value was found to be higher than its bulk value. According to the researchers, Ni NPs in HT29 cell lines is also sphere-shaped and poisonous. *Medicago sativa*, or alfalfa plant extract, made Ni NPs.[33] Nickel bio-reduction was initiated by flavonoids and reducing sugars in the extract. Biosynthesis, they argue, is a more economical and promising substitute for traditional nickel nanoparticle manufacturing procedures. A plant extract from *Annona squamosa* (sugar apple) was used to make Ni NPs.[34] The arylamine in the phytochemical of the leaf extract, according to their findings, is responsible for converting Nickel oxide to Ni NPs. *Aegle marmelos* is an eagle species. Correa plant leaf extract was used as a reducing, stabilizing, and capping agent in the synthesis of Ni NPs.[35] Anti-inflammatory and larvicidal mosquito efficiency of Ni NPs and *Aegle marmelos* Correa crude leaf extract was also investigated in vitro against three blood-feeding parasites. Ni NPs outperformed crude leaf extract in terms of anti-inflammatory and larvicidal activity.

4.3 Using starch and plant secretion as a reducing agent

To make NiO NPs from Arabic gum, Fardood et al. (2017) used a solvent–gel technique. They revealed that they used Arabic gum gel as a biopolymeric template to make the first green NiO NPs (36). The process, they suggested, could be used to make low-cost transition metal NPs from their oxides and other materials produced Nano-sized nickel material using starch in several additional works on generating Ni NPs using green agents. Carbonising starch and the metal salt is flowing hydrogen formed peculiar core-shell-structured Ni NPs. It looks to be a novel method for producing carbon-encased metal NPs.[37]

4.4 Microorganisms as a reducing agent

Dias et al. (2002) employed *Aspergillus terreus* cells embedded with polyurethane foam.[38] They demonstrate that bio-absorbents can replace synthetic adsorbents in industrial waste treatment. Bio absorbents are considerably less expensive than synthetic resins and may be recycled. Algae, both alive and dead, have been demonstrated to convert noble metals to metal NPs in the literature.[39] It opens up the prospect of employing organisms to synthesize metal NPs in an environmentally benign manner and making them from microbes.[40-42]

According to these studies, green synthesis employing plant parts has betterment over the past ten years. The literature is constantly updated with new plant extracts that can be used as reducing agents in the manufacture of Nickel NPs. Plant synthetic techniques were eco-friendly and made use of low-cost starting materials.[43] According to the report, green reducing agents were used to produce Ni NPs. Except for *Annona squamosa* and *Chlorella vulgaris*, all plant-assisted NPs were sized between 1 and 100 nanometers. As a result, they can catalyze a variety of processes.

Compared to chemically generated Ni NPs, green-synthesized Nickel NPs were initiated to have consistent antioxidant and antimicrobial activities and are harmless to animal cells. Ni NPs made using *Aegle marmelos* Correa leaf extract as a reducing agent had better anti-inflammatory and larvicidal properties than pure leaf extract. HT-29 colon tumor cell lines were used to examine the cytotoxic action of Ni NPs generated by

Azadirachta indica and *Psidium guajava*. When the cells were treated with the created Nickel Oxide and Ni NPs, essential variations in cell morphology, such as swelling and cell disintegration, were observed. Bio absorption studies using *Aspergillus terreus* to generate Ni NPs revealed that this strain of A. *terreus* can be a good candidate for industrial application for removing iron, chromium, and Ni NPs due to its affinity for binding a mixture of 3 weighty metals in solution simultaneously. New plant extracts as reducing agents in the synthesis of Ni nanoparticles were published in the literature regularly. Numerous creatures have also been discovered to manufacture Ni NPs and open up the possibility of environmentally friendly metal NPs produced from plant extracts and organisms.

4.5 Bacterial synthesis of NiO NPs

Because these industries produce carcinogenic nickel (II) ions, their concentration in industrial effluents must be decreased or eliminated. For example, Ni was commonly used in steel, batteries, and electroplating sectors. Unfortunately, traditional waste-water treatment systems are ineffectual and expensive because they typically discharge secondary contaminants into the environment. As a result, it is a significant challenge to develop cost-effective and environmentally friendly treatment technologies capable of removing soluble nickel (II) ions from manufacturing effluent or converting them to a soluble divisible form, as Nickel Oxide is insoluble in water.

Satyavati et al. converted soluble $NiSO_4$ detected in discharged electroplating effluents to insoluble NiO. (2014). (Sewage diluted with NaCl was plated on nutrient agar and incubated at 30°C for bacterial growth.) The bacteria were cultivated for 120 hours at 200rpm in industrial effluent containing 2172mg/L Nickel (II), then centrifuged to remove the bacterium cells. The pale green sediments built up in the bottom of the flask were removed, washed, dried, and analyzed. The function of metabolism-dependent and physiologically controlled mechanisms in the nucleation and deposition of inorganic elements and the efflux mechanism, which involves extracellular Nanomaterial formation, are investigated in the production of metal oxide Ni NPs. The sediments were NiO NPs with a flower-like morphology and a 100nm–50nm size range. As a result, this technology offers a green route for the remediation of hazardous soluble Nickel (II) ions, with a nickel removal efficacy of 95%.[44]

4.6 NiO NPs synthesis using Fungi

Equally alive and lifeless fungal biomass can be used to create nanoparticles. Lifeless fungal biomass has the advantages of being stored for longer periods, requiring no nutrients or growth media, and being low in toxicity. One of the advantages of using fungi to make green NPs is that optimal mycelia development can replenish a large surface area. Furthermore, by adsorbing metal ions on the cell wall, which contains chitosan, chitin, amino group phosphates, glucan, lipids, sulphates, phospholipids, and hydroxides, among other things, fungal biomass may be able to tolerate metal toxicity. These functional groups act as metal bioabsorption binding sites.[45] Toxic metal poisoning of natural water bodies and sediments is one of the century's most pressing problems. Because of increased smelting and mining activity over the last decade, Nickel has grown more abundant in the environment. The interactions of microorganisms with metals are well understood, and the capacity of bacteria to acquire and remove metals is already being used in bioremediation via hazardous metal bioabsorption. However, the mechanism underlying this behaviour remains unknown. Fungi are great candidates for metal bioabsorption because they can live in a wide range of temperatures, pH levels, nutrition levels, and high metal concentrations.[46]

Salvadori and colleagues used NiCl as a precursor and lifeless, dried, and alive biomass of the filamentous fungus *Aspergillus aculeatus* (MIC = 2000mgL1) as a reducing agent to make Nickel Oxide nanoparticles. Lifeless fungal biomass had the highest adsorption capacity and thus the best resistance to metal toxicity amongst the 3 forms of fungal biomass utilized in this test. After the requisite biomass and precursor solution doses were shaken at acceptable reaction conditions, the Nickel (II) solution was vacuum filtered from the biomass using Millipore membranes. The Nickel Oxide nanoparticles that were obtained were investigated using various methods. Proteins were detected on the Nickel Oxide nanoparticle's surface in the form of a film that might serve as a capping agent using energy-dispersive X-ray spectroscopy.

The same group of researchers created NiO NPs via bioabsorption of Nickel (II) ions on the living, drying, and dead biomass of the *Hypocrea lixii* fungus (MIC = 1473mgL1). According to the maximum Nickel (II) retention capacity observed in dead biomass, excessive metal concentrations might cause dried and live biomass toxicity. In Transmission Electron Microscope and High-Resolution Transmission Electron Microscope micrographs of dead biomass loaded with NiO NPs, nanoparticles were identified on the fungal cell wall both extracellularly and intracellularly via bio-absorption. Micrographs demonstrate that the autoclaving method generated substantial structural alterations in the Nickel Oxide-loaded dead biomass and the control sample, including cytoplasm shrinkage. Extracellular and intracellular NiO NPs took an average diameter of 3.8 and 1.25nm, respectively, and were both spherical. Nickel (II) adsorbs to the fungal cell wall via its amide group, converted to metallic Nickel in a two-step process by fungal cell wall enzymes. H2O and O2 oxidized Nickel to Nickel Oxide in the medium in the second stage. Fungal cell wall proteins/peptides were used as a capping agent for the created NPs. The EDX pattern of NiO NPs revealed new C, N, and O peaks. In contrast, FTIR analysis revealed that the band for N-H of amide II linkages of proteins/polypeptides at 1535cm1 had shifted to 1542cm1, indicating that the N-H group had twisted as it capped the NiO NPs.[47]

The researchers used nickel nitrate hex hydrate as a precursor and Rhizop as a reducing and stabilizing agent to create NiO NPs. The fungus was coarsely chopped and removed from the bread before being added to the precursor solution. 1M NaOH solution was used to change the pH of the solution. After stirring for 5 hours, the solution was kept overnight and filtered through Whatman filter paper. After that, the filtrate was calcined for 5 hours at 500°C, providing a sample crushed into minute particles. Polydisperse NPs with an average diameter of 40nm were discovered during the characterization of NiO NPs. Because these NPs are polydisperse, monodisperse NPs can be made by adjusting experimental parameters, including pH, temperature, and the number of reductants and precursors used.[48]

5. Characterization of NiO NPs

UV-Vis spectroscopy, X-ray diffraction (XRD), Fourier transformation infrared spectroscopy (FTIR), transmission electron microscopy (TEM), scanning electron microscopy (SEM), zeta potential measurement, thermogravimetric analysis (TGA), X-ray photoelectron spectrometry (XPS), photoluminescence (PL), and energy-dispersive X-ray spectroscopy (EDXRS) are examples (EDS).

Ni NPs exhibit a surface plasmon resonance (SPR) absorption band between 300 and 400 nanometers, making them plasmonic. SPR arose when metallic NPs physically absorbed light, causing the metal's conduction electrons to oscillate coherently. The amplitude of oscillation reaches its maximum when the frequency of an incident photon equals the natural frequency of surface electrons, and this frequency is referred to as SPR. The light absorption is measured using a UV/VIS spectrophotometer.[49]

The biomolecules responsible for turning metal salt into MNPs and their subsequent growth inactivation due to the capping effect of these biomolecules are investigated using FTIR spectroscopy. For this, we used the FTIR spectra of plant extract and MNPs. The biomolecules involved in bioreduction should only be present in the extracted spectrum and disappear in the NPs spectrum. It helps identify biomolecules involved in bioreduction and creates a reaction mechanism.[50] Recording the FTIR spectrum of plant/microbial extract earlier and afterward bioreduction is another technique to recognize the biomolecules responsible for bioreduction. After bioreduction, the band soft hose biomolecules responsible for bioreduction alter their placement in the examined spectrum.[51]

Using the XRD technique, researchers may identify, purify, and quantify NPs. The value of crystal planes for a specific nanoparticle is calculated by recording the peaks at 2 values; these peaks define the value of crystal planes for that nanoparticle. The NPs and their phase can be recognized by comparing the position and strength of these diffraction peaks to the JCPDS card number (each type of metal has its own JCPDS card number) (spherical, wurtzite, etc.). Particle crystallinity has an impact on the strength of XRD spectrum peaks. When NPs' crystallinity is high, they exhibit intense and sharp peaks and vice versa. Use an equation to fill in the gaps. The Scherrer equation can also be used to determine the size of NPs; when the particle size is large, the XRD patterns become broad. D=0.98λ /βcosθ where D stands for particle size, CuK for wavelength, FWHM for full width at half maximum, and diffraction angle for diffraction angle.[52]

SEM scans the surface of NPs with a high-energy electron beam created by a heated filament to determine their form and content.[53] The TEM is used to determine the interior composition of NPs, such as form, size, size distribution, and flaws.[54]

6. Biomedical applications of Ni NPs

Ni NPs have been used in biological applications and as an antibacterial agent in the literature. Examples are drug and gene delivery, magnetic resonance imaging, cell separation, biomedical detection, and diagnostics.[55] Functionally charged Ni NPs, according to Guo et al., can improve cell membrane permeability and increase cellular absorption of foreign target chemicals into cancer cells (2008). These results suggest that Ni NPs have a mechanism for decreasing cytotoxicity in leukemia cancer cells. Ni NPs have the potential to be used in a variety of biological and medicinal applications. According to Ivanov et al. (2018), the synergistic effect of Ni NPs with reduced graphene oxide permits the creation of high-performance applications in bioinspired microelectronics for medical therapy.[56] According to Angajala et al., Ni NPs show a high larvicidal effect against *Culex quinquefasciatus* and provide significant anti-inflammatory benefits comparable to conventional treatment (2014). They hypothesized that Ni NPs might be utilized to inhibit C.*quinquefasciatus*, a vector for lymphatic filariasis and dengue fever transmission. Ni NPs, according to Helan et al. (2016), could be used to minimize microbiological infections such as Staphylococcus aureus and E. coli.[57] Ni NPs have outstanding catalytic and magnetic capabilities, showing their enormous potential in a variety of applications, including biological and medical domains, according to Roselina and coworkers. According to Gong et al. (2014), Algae can speed up NPs aggregation and reduce NiO to Nickel. Green algae, they argued, could be effective in Nanopollution bioremediation. Green Ni NPs, according to Sudhasree et al. (2014), have colloidal stability and antioxidant properties, making them efficient antibacterial agents.[58]

7. CONCLUSION

This research aims to look at how plant extracts, microbial extracts, and naturally occurring proteins can be used to manufacture Ni and NiO in a greenway. Plant extracts are affordable, environmentally friendly, and adaptable.

On the other hand, traditional chemical synthesis methods needed many reactants, lengthy procedures, and complicated apparatus. Our green methods for generating nickel and NiO NPs, on the other hand, were simple, efficient, and environmentally benign. Green chemistry is an excellent alternative for nickel and nickel oxide nanoparticle synthesis because of its environmental friendliness, economic prospects, feasibility, enhanced biocompatibility, low cytotoxicity, and exceptional antioxidant and antibacterial activity. Ni and NiO NPs can be used in environmental remediation and nanomedicine because of their properties.

Ni NPs produced biogenically are free of harmful compounds and suitable for biomedical uses. Therefore, Ni NPs have been widely investigated in their manufacture, characterization, and applications. New green approaches will be employed in the coming days to create Ni and NiO NPs with various structural properties and biological effects.

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