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Review

Nickel Oxide Based Nanoparticles: Green Synthesis, Morphological Assessment, and Their Biological Properties

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Abstract: Nanotechnology has been successfully implemented for improved applications in biological activities and environmental remediation. Nickel oxide nanoparticles (NiO-NPs) are effectively synthesized from bio-inspired sources, and their effectivity is higher than that of chemically synthesized NiO-NPs. Biogenic synthesis is mostly favored due to its low cost and eco-friendliness. Properties of NiO-NPs, such as wide bandgap, semi-conductivity, good electrochemical performance, etc., have proved to be superior to those of other Metal Oxide Nanoparticles. Phytochemicals present in various medicinal plant materials act as chelating agents, capping agents, and stabilizers for the green synthesis of NiO-NPs. While various organic molecules present in microorganisms reduce metal salts to Metal Oxide Nanoparticles as they act as reductants or dispersants, distinct characterization techniques such as XRD, SEM, TEM, UV-VIS, FTIR, EDS, etc. are used to confirm the formation of NiO-NPs and for their morphological studies. Green chemistry is witnessing rapid progress by virtue of its ease of synthesis, inexpensiveness, non-toxicity, and renewability. NiO-NPs have a wide range of applications. Biochemical applications such as antifungal, antibacterial, antidiabetic, anticancer, and anti-larvicidal are discussed with their plausible mechanisms. Photocatalytic activities of NiO-NPs are also studied in detail, in which a light-activated catalyst is used for the reduction or oxidation of organic dyes. The present review systematically represents various routes for green synthesis of NiO-NPs, morphological elucidation through well-known quantitative techniques, and potential applications with respect to catalytic and biological fields.

Keywords: nickel oxide nanoparticles, biogenic synthesis, morphological studies, biochemical applications

1. Introduction

Nanotechnology is modernizing the technological and industrial fields due to the distinct properties of nanoparticles. Materials show different physiochemical properties and new effects at the nano level compared to the corresponding macro level due to the increase in surface-to-volume ratio with the reduction in size of nanoparticles. Other properties, like catalytic, thermal, and mechanical, of the material change due to modifications in the ratio of the surface area to the volume [1–3]. By using nanotechnology, the size, shape, and morphology of materials can be manipulated. Thus, making nanoparticles lighter, stronger, more reactive, and more durable, and hence altering their properties so as to design

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them for use in different fields, viz., pharmaceutical, medical, environmental, electronics, mechanical industries, etc. [4,5]. Nanotechnology is a branch of science and engineering that deals with designing, producing, and manipulating atoms and molecules at the nanoscale, i.e., less than 100 nm. The names of nanoparticles are based on their diverse shapes and structures. They are termed nanoflower, nanocube, nanowire, nanotube, cluster, core shell bimetallic, etc. based on their shapes and structures, respectively. While they are also termed cluster, core shell bimetallic, etc. based on their structure. There are many methods of synthesis of nanoparticles (chemical, physical, biological, and hybrid methods), of which chemical and physical methods are popular. But chemical or physical methods involve some limitations, which involve the generation of a large number of hazardous by-products that are highly concerned with environmental contamination. Hence, there was a need to develop nontoxic, efficient, and eco-friendly methods. The biogenic method involves nanoparticle synthesis by using microorganisms (bacteria, yeast, and fungi) and plant parts (leaves, stems, roots, bark, flowers, fruits, and seeds) [6–8].

Metal nanoparticles are synthesized in two forms by the microorganism, i.e., outside the cell and inside the cell. Organic molecules present in microorganisms act as reductants or dispersants. Though it is not evidently clear which organic molecules act as reductants or dispersants. The extracellular components act as dispersants in the process where metal nanoparticles are synthesized outside the cell. With the extracellular components and the added electron donor, membrane components behave as reductants. Proteins, or peptides, function as dispersants in the synthesis of metal nanoparticles inside the cell, wherein the cytoplasm behaves as a reductant on account of its electron-donating tendency to metal ions [9,10].

Plant extracts act as reducing, stabilizing, and capping agents and are used to synthesize metal oxide NPs. Different parts of plants (edible and inedible) are used for the synthesis of metal oxide NPs. Secondary metabolites such as carbohydrates, proteins, and coenzymes reduce metal salts into NPs. The extraction of plant extract is simple and easy to store. The metal salts have a high reduction potential, and the metal attached to them is electron-deficient due to their tendency to donate electron density to conjugative salts. Hence, metal ions get reduced by plant extracts, which act as reducing agents [11–13].

To date, researchers have synthesized various Metal and Metal Oxide Nanoparticles such as titanium dioxide (TiO_2), silver (Ag), silver oxide (AgO), iron (Fe), iron oxide (FeO), gold (Au), copper (Cu), copper oxide (CuO), zinc (Zn), zinc oxide (ZnO), platinum (Pt), etc. Out of all these Metal and Metal Oxide Nanoparticles, nickel (Ni) and nickel oxide (NiO) nanoparticles have spellbound scientists due to their superior properties. They are easily synthesized, smaller in size, have a wide bandgap, and have semiconductor properties [14]. Scientists have studied the electrochemical properties of MgO, RuO, CuO, TiO, FeO, and NiO. Among other metal oxides, NiO has shown good electrochemical performance. NiO has a band gap ranging from 3.6 to 4.0 eV, and it is a stable p-type semiconductor. NiO nanoparticles have applications in batteries, sensors, superconductors, magnetic materials, and photocatalytic and catalytic analysis [15].

The characterization and morphological analysis of synthesized Ni and NiO nanoparticles are deliberated by using UV-Visible spectroscopy (UV-Vis), Fourier transformation infrared spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), and Energy Dispersive X-ray Spectroscopy (EDS) [2,7]. NiO and Ni NPs have numerous biomedical applications. It has enhanced antibacterial, antifungal, anti-larvicidal, anti-cancer, and antidiabetic activities. Several studies have shown antibacterial activity against *Escherichia coli*, *Escherichia hermannii*, *Pseudomonas aeruginosa*, *Proteus vulgaris* (gram-negative), *Bacillus sublitis*, *Streptococcus pneumonia*, and *Staphylococcus aureus* (gram-positive) bacteria, as well as antifungal activities against *Candida albicans*, *Mucor racemosus*, *Aspergillus niger*, *Aspergillus fumigatus*, *Fusarium solani*, *Aspergillus flavus*, *Alternaria alternate*, *Penicillium* spp., and *Fusarium* oxysporum. Advance engineering has been used to develop drug delivery systems incorporated with Ni/NiO-NPs to treat site-specific treatment against infections or related diseases. Ni/NiO-NPs, due to their particle size distribution, surface area, volume, and shape, show effective results. Further, the antioxidant properties of Ni/NiO-NPs play a vital role in stabilizing free radicals, harmful for human cells. Reports show that Ni/NiO-NPs donate electrons to free radicals, making them less reactive without getting destabilized [16–20]. Many anti-cancerous tests for NiO and Ni NPs have been successfully carried out against MCF-7 cancer line models, PC12 cell line, normal nervous neurospheres (NCs) cell, and human glioblastoma cancer (U87MG) cell line. Apart from biomedical applications, Ni/NiO-NPs

additionally show photocatalytic activity, dye-sensitized solar cells and sensors, Dye adsorption from industrial waste and catalytic activities [21–25].

Herein, we discuss the various routes for the biosynthesis of Ni and NiO nanoparticles from plants and microorganisms. Plausible interactive mechanistic details of biomedical applications, such as antibacterial, antifungal, anti-larvicidal, anticancer, and antidiabetic activities are discussed thoroughly. Additionally, dye degradation by photocatalysis, use of Ni/NiO-NPs in catalytic reactions, dye adsorption from industrial waste, and dye-sensitized solar cells are also explained with a proper explanation.

2. Method of synthesis of Ni/NiO-NPs

Preparation techniques involved in Metal Oxide Nanoparticles (MO-NPs) synthesis include two different approaches. Principally, MO-NPs can be synthesized using a top-down and bottom-up approach. Basically, top-down synthesis involves the decomposition of bulk material into nanoparticles [26,27]. It comprises the thermal decomposition method, mechanical milling or ball milling, lithography, laser ablation, and sputtering, whereas bottom-up synthesis includes chemical vapor decomposition, the sol-gel process, spinning, pyrolysis, and biological synthesis. The method of synthesis where various parts of plant extract and microorganisms are involved is termed biological, biogenic, or green synthesis. The simple and eco-friendly method where plant parts such as roots, stems, leaves, bark, flowers, and fruit are used for MO-NPs synthesis involves the reduction of metal ions into Metal Oxide Nanoparticles. The phytochemicals, such as alkaloids, flavonoids, terpenoids, polysaccharides, and heterocyclic compounds, play a vital role in the reduction process. The microbial synthesis involves metal ion reduction by reductase enzymes present in microorganisms, while proteins, reducing cofactors, metal-resistant genes, enzymes, and organic material act as capping and reducing agents. Microbes such as fungi, yeast, and bacteria are used for the MO-NPs synthesis [28,29].

2.1 *Plant-extract mediated synthesis of Ni and NiO nanoparticles*

Plant extract-mediated synthesis of one- or multi-dimensional particles with a size less than 100 nm has gained much attention in the last 30 years due to its low cost and eco-friendly process [30]. Plant parts such as leaves, stems, roots, bark, fruits, and flowers are widely used for the preparation of nickel and nickel oxide nanoparticles, as shown in Figure 1.

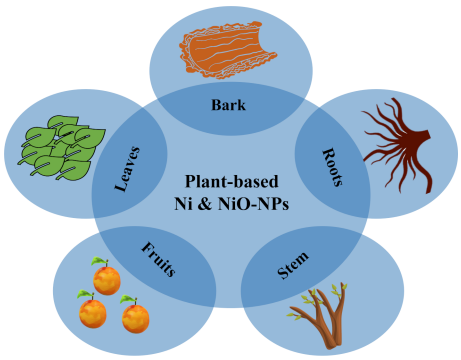

Figure 1. Diagrammatic representation of plant parts used for NiO-NPs preparation

Naturally occurring phytochemicals such as phenolic compounds, alkaloids, flavonoids, polyphenols, etc. help in the reduction of nickel salt into Ni and NiO nanoparticles and act as stabilizing and capping agents. Capping agent has a polar head and a non-polar hydrocarbon tail; hence, it is known as an amphiphilic molecule. Both ends of capping agents interact with their surroundings in different ways [31]. The polar end interacts with the metal ion of the nanoparticle, while

the non-polar end interacts with its surroundings, as shown in Figure 2. The synthesis in which plants are used as capping and reducing agents for nanoparticle fabrication is termed green synthesis.

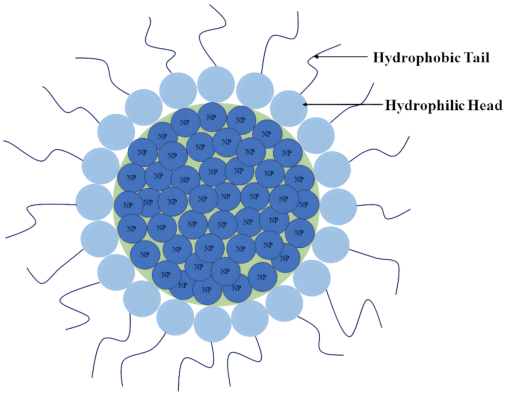

Figure 2. Covalent interaction between NPs and capping agents

There are different types of capping agents, which include polyethylene glycol (PEG), polyvinylpyrrolidone (PVP), polyvinyl alcohol (PVA), bovine serum albumin (BSA), ethylenediaminetetraacetic acid (EDTA), chitosan, and phytochemicals. All the different types of capping agents function similarly with respect to controlling size, stabilizing, and protecting the morphological characteristics of nanoparticles. But phytochemicals are preferred over all other types of capping agents because these conventional surfactants are toxic and very tough to remove [32]. Hence, there is a need at the commercial and non-commercial levels to use eco-friendly capping agents. Moreover, they enhance the functionality of synthesized nanoparticles by slightly changing their properties. Variations like reduction of toxicity, biocompatibility enhancement, and bioavailability are made by capping or stabilizing agents to enhance the biomedical functioning of nanoparticles. Cluster formation or aggregation is one of the prime limitations of the nanoparticle formation process. Due to nanoparticle aggregation, the problem of uncontrolled growth and colloidal stability is affected. Capping and stabilizing agents play an important role in overcoming this limitation [33,34]. Plant-based phytochemicals act as capping and stabilizing agents and prevent clusters or aggregation of nanoparticles, as shown in Figure 3. Metal ions are reduced and stabilized by the phytochemicals present in plant extracts. These phytochemicals operate through the following mechanisms: (i) Formation of intermediates of varying oxidation states with metal ions in them. (ii) served as capping agents for the non-cluster framework of nanoparticles. The findings reveal that morphological assessment and biological properties depend on the volume and nature of the extract.

The secondary metabolites act as reducing agents and reduce metal ions. They donate electrons, forming a coordination bond between −OH of phytoconstituents and metal ions and hence acting as a chelator of metal ions. Further reduced metal ions undergo nucleation, followed by the spontaneous coalescence of small NPs into larger NPs. The final step is oven drying and calcination of the product to form nanoparticles [35]. The synthesis of NiO-NPs by using different plant parts extracts is discussed as shown in Table 1.

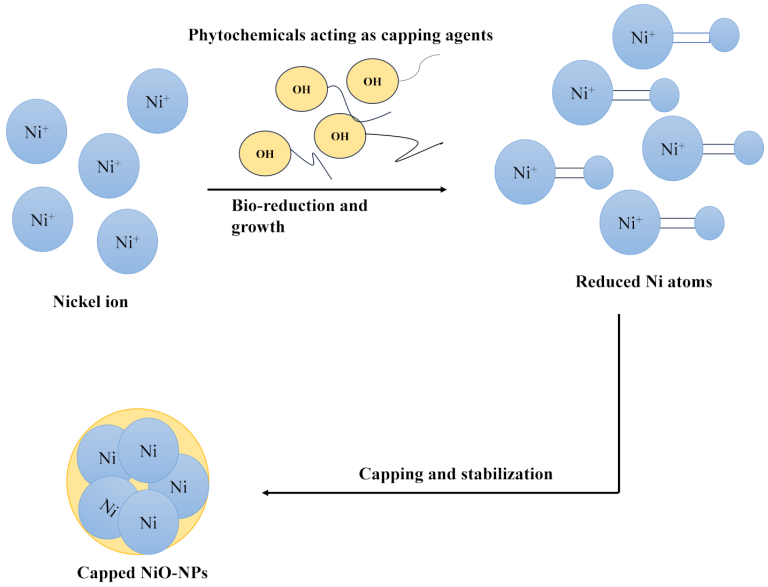

Figure 3. Bio-reduction, capping, and stabilization of NiO-NPs

Helan et al.[36], reported the synthesis of FCC NiO-NPs by reducing $NiCl_2$ (Aq.) with an aqueous extract of Neem leaves. Synthesized NiO-NPs were subjected to XRD, FTIR, SEM/EDAX, and TEM analysis for their structural and morphological studies. Leaf extract of the Okra plant was prepared by Zahra Sabouri et al.[37], for NiO-NPs synthesis. They reacted Okra leaf extract with a $Ni(NO_3)_2{\cdot}6H_2O$ solution by stirring in an oil bath for 6 h at 80 °C on a magnetic stirrer to obtain sticky green-colored gel. It was dried in an oven and calcinated to obtain NiO-NPs. Gebretinsae et al.[38], used the dry leaves of *Opuntia ficus indica* for NiO-NPs synthesis. The structural analysis using XRD shows cubic NiO-NPs with an average crystalline size of 16.01 nm.

Nasseri et al.[39], synthesized NiO-NPs using an aqueous flower extract of *T. serotina*. The extract was prepared by mixing dried flower powder with deionized water as a precursor. Further, the flower extract was reacted with a $Ni(NO_3)_2{\cdot}6H_2O$ solution at 115 °C for 1.5 h in an oil bath, followed by drying and calcination at 400 °C for 1 h. Finally, spherical-shaped NiO-NPs of 10–14 nm were synthesized. Hussein et al.[40], prepared the fruit extract from grapes by crushing it in 250 mL of deionized water, followed by filtration through a Whatman filter paper No. 1 to obtain a light-yellow transparent solution. Grape's extract was reacted with 0.1 M nickel (II) chloride hexahydrate ($NiCl_2{\cdot}6H_2O$) in a 1:1 ratio. The color change from light green to dark green indicates the NiO-NPs formation and is further confirmed by standard characterization techniques like SEM, TEM, AFM, XRD, and FT-IR. Ezhilarasi et al.[41], used *Phoenix dactylifera* (Dates) extract and reacted it with a 0.1 M $Ni(NO_3)_2$ solution to synthesize NiO nanorods. The bark extract of *Spirostachys africana* was reacted with a $Ni(NO_3)_2{\cdot}6H_2O$ solution and allowed to stir for 4 h. Further, the reacting mixture was dried in an oven, followed by calcination at 500 °C for 2 h[42].

Table 1. Plant parts, respective precursors used for the synthesis of Ni/NiO-NPs, their shapes, sizes, and applications

Plant (Part)	Precursors	Characterization Techniques	Shape of NPs	Size of NPs (nm)	Crystalline Structure	Application	Ref.
Neem (Leaf)	$NiCl_2$	XRD, TEM, FTIR, EDAX	Hexagonal	10 ± 2	FCC	Antibacterial	36
Okra (Leaf)	$Ni(NO_3)_2$	UV–Vis, FTIR, TGA/DTA XRD, EDS, VSM, FESEM	Spherical	18.6	FCC	Cytotoxicity, Antibacterial	37
Cactus (Leaf)	$Ni(NO_3)_2$	XRD, SEM	Spherical	20–35	Cubic	-	38
Tamarix serotina (Flowers)	$Ni(NO_3)_2$	TEM, XRD, UV-Vis, VSM, BET	Spherical	10–14	Cubic	Catalytic activity	39
Vitis vinifera (Fruit)	$NiCl_2$	SEM, TEM, XRD, AFM, FTIR, UV-Vis	Spherical	10–30	Cubic	Anticancer, Antibacterial	40
Phoenix dactylifera (Fruit)	$Ni(NO_3)_2$	XRD, FT-IR, HR-TEM, UV-Vis	Rod	32 ± 5	FCC	Antibacterial, Photocatalytic degradation, Cytotoxicity	41
Spirostachys africana (Bark)	$Ni(NO_3)_2$	FT-IR, SEM, EDS, XRD	Amorphous	-	-	-	42
Alfalfa	$Ni(NO_3)_2$	TEM, XRD, XPS, FT-IR	Spherical	3.4 ± 0.9	FCC	-	43
A. marmelos Correa (Leaf)	$NiCl_2$	UV-Vis, IR, TGA, XRD, SEM, AFM	Triangular	80–100	FCC	Anti-inflammatory, Anti-larvicidal	44
Moringa oleifera (Flowers)	$Ni(NO_3)_2$	HRTEM, XRD, EDS, FTIR	Cylindrical and Spherical	9.69		Antibacterial, Anticancer	45

2.2 *Microorganism-based synthesis of Ni and NiO NPs*

Microbes such as fungi, bacteria, and algae are used for the fabrication of Ni and NiO nanoparticles due to their reducing and stabilizing properties, as shown in Figure 4. Reductase enzymes, proteins, reducing cofactors, metal-resistant genes, and organic material in microorganisms act as reducing and stabilizing agents.

Utilization of fungi for the synthesis of MO-NPs has become a very common technique as it has many advantages, including economic feasibility, ease of processing, and handling of biomass for optimum growth of mycelia. The fungus-mediated green chemistry is preferred over other microorganisms as they are easy to use, their cell wall-binding capacity is higher, they have high intracellular metal uptake capabilities, and they require simple nutrients. A biosorption mechanism through the cell wall is used by fungi against metal toxicity. Functional groups such as carboxyl groups, amino groups, phosphates, lipids, melanin, sulfates, and hydroxides are present in chitin, chitosan, glucan, lipids, and phospholipids of fungal biomass walls and are the main reason for metal sorption. Salvadori et al. [46], has used dead *A. aculeatus* biomass for NiO-NP synthesis through biosorption. It is reported that *A. aculeatus* biomass has a higher adsorption capacity (19.6 mg/g) than other biosorbents. Salvadori et al. [47], have proposed possible extracellular and intracellular mechanisms involved in the synthesis of NiO-NPs from the dead biomass of *H. lixii*. In the first of two steps of the extracellular mechanism, amide groups present in the cell wall interact with Ni^{2+} and reduce them to metallic Ni with the help of enzymes also found in the cell wall. The extracellularly produced NiO-NPs have enormous secretory components that help in the process of biological reduction and act as stabilizing and capping agents to prevent agglomeration, as per FTIR analysis. In the second step, the Ni-NPs formed are oxidized by H_2O and O_2 already present in the medium due to the fact that nickel has a negative reduction potential. Thus, the mechanism of NiO-NPs formation by using fungal biomass involves reduction of metal ions and extra- and intracellular oxidation of metal nanoparticles to MO-NPs. The intracellular mechanism, similar to the extracellular mechanism, involves Ni^{2+} ion interaction with the fungal cell wall through electrostatic interaction with the enzymatic groups of the mycelial cell wall. This interaction probably leads to a reduction of Ni^{2+}, followed by aggregation to form NiO-NPs. The major difference between extracellular and intracellular mechanisms is the size of the MO-NPs formed.

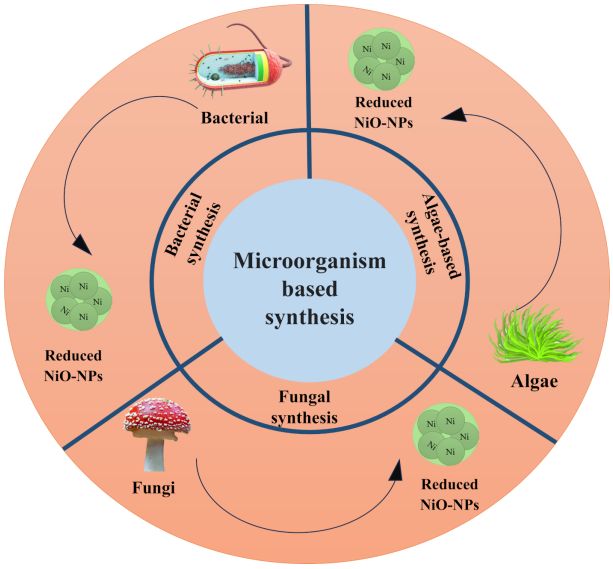

Figure 4. Microbial synthesis of metal and MO-NPs

Nanoparticles formed are smaller in intracellular mechanisms than extracellular mechanisms. Another major difference is the way nickel oxide NPs are separated from the biomass. The extracellular NiO-NPs are separated from biomass by centrifugation technique, and the intracellular NiO-NPs are separated by ultrasound treatment of biomass or by reacting it with suitable detergents. Atalay et al.[48], synthesized nickel oxide porous microtubes by the chemical precipitation method using the *C. cladosporioides* fungus. The given strain of fungus was grown in Potato Dextrose Broth (PDB), and the obtained mycelial biomass of the *C. cladosporioides* fungus was reacted with $NiCl_2$ and NH_4OH solutions. El-Debaiky et al.[49], have also reported the fungi-based preparation of NiO-NPs. For this purpose, biomass and mycelial filtrate of *F. verticillioides* were grown in PDL medium and reacted with 0.001M $Ni(NO_3)_2{\cdot}6H_2O$ solution. The obtained product in the form of ppt was air dried, heated at 200 ^oC for 12 h, and annealed at 400 and 600 ^oC for 2 h to obtain NiO-NPs. The algae-mediated synthesis of MO-NPs has been categorized into three major techniques, which include the direct manipulation of algae cells, the breakdown of algal cells, followed by extraction using different downstream process procedures such as filtration and centrifugation, and using the supernatants of the algal broth for the harvesting of nanoparticles. Bio-active components like polyphenols and tocopherols (antioxidants), carotenoids (pigments), chlorophylls and phycobilin, vitamins, carbohydrates, oil, nutrients, fats, and polyunsaturated fatty acids are found to act in different concentrations as reducing and stabilizing agents[50].

Bacteria-mediated synthesis is a simple and eco-friendly technique used for MO-NP synthesis. Bacteria are prokaryotic microorganisms found abundantly in soil, water, and air, with diversities in shapes like bacilli (rods), cocci (spheres), and spirochaetes (spirals). Bacteria adapt themselves easily to harsh conditions, grow easily, and can also be cultivated inexpensively. By using different bacterial strains, researchers have synthesized MO-NPs with different morphologies. Against the effects of heavy metals, bacteria use remediation techniques to reduce heavy metal toxicity, thus producing nanoparticles. MO-NPs synthesized in either unicellular or multicellular bacteria follow a general scheme in which bacterial cells capture metal ions. The bacterial synthesis of MO-NPs is similar to fungi-based synthesis. Both involve intracellularly and extracellularly the formation of MO-NPs, depending on the localization site. Initially, metal ions interact with bacterial cells and enter through endocytosis, active transport, or forcefully penetration of the lipid membrane. In the process of intracellularly synthesized MO-NPs are synthesized inside the cell, where the processes of trapping, bioreduction, and capping of metal ions are accomplished inside the cell wall. Extracellular synthesis is similar to intracellular synthesis, which includes secretion of enzymes, bioreduction, and particle capping. Extracellular mechanisms are preferred over intracellular mechanisms due to high efficiency and easier purification and extraction. Chemical parameters like organic functional groups and physical parameters such as temperature and pH are some of the several factors responsible for

the reduction of nanoparticles. The variations in physical parameters like pH and temperature conditions also affect the morphology, i.e., shape and size of MO-NPs. Additionally, the characteristic properties are more distinct due to the small size of the nanoparticles. Hence, there is a great scope in bacterial-mediated Ni and NiO-NP synthesis, its characterization, and studying the enhancement in biological applications[51–55].

3. Characterization techniques

NiO nanoparticles can be synthesized biogenically by using plant parts and microorganisms. It is a suitable, easier, simpler, and eco-friendly synthesis procedure. Synthesized NiO nanoparticles can be characterized by using the following techniques: Synthesized NiO-NPs are characterized to study their shape, size, morphology, chemical composition, and various properties[56]. Characterization techniques extensively used are discussed in Figure 5

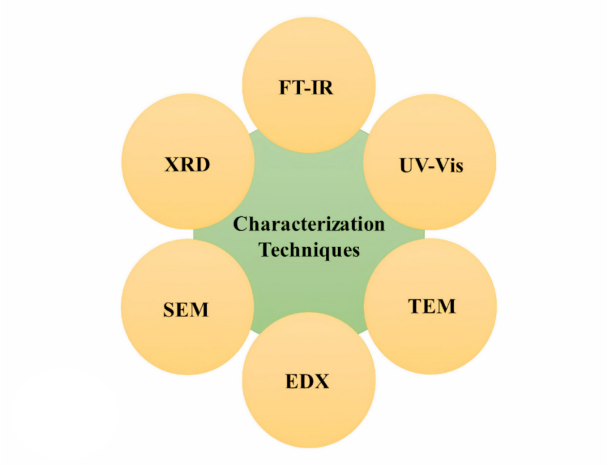

Figure 5. Standard analytical techniques used for the characterization of nickel and nickel oxide nanoparticles

3.1 *Fourier Transform Infrared Spectroscopy (FTIR)*

Fourier Transform Infrared Spectroscopy (FTIR) helps study the vibrations of functional groups associated with nanoparticles. Phytochemicals present in plant extracts are responsible for the reduction of metal ions into Metal Oxide Nanoparticles. Hence, to investigate the phytochemicals responsible, the FTIR characterization technique is extremely useful. This technique is a non-destructive one, allowing fast and precise determination of ligands associated with nanoparticles[50,57].

Olajire et al.[15], have carried out FTIR analysis to study functional groups in phytochemicals responsible for the reduction, capping, and stabilization of plant-mediated synthesized NiO-NPs. The aqueous leaf extract of *A. comosus* was analyzed, and a broad peak for the O-H bond (3308 cm^{-1}) and a weak band for the C-H bond (2852 cm^{-1}) were found. They also analyzed the presence of a triple bond of C-N or C-C (2135 cm^{-1}). The FTIR spectrum of *A. comosus* leaf extract found 1734 cm^{-1} (>C=O), a sharp peak of 1608 cm^{-1} (>C=C<) of aromatic rings, in-plane O-H bending (1406 cm^{-1}), C-O stretch (1265–1064 cm^{-1}) of polyols, and C-H bend (918–623 cm^{-1}) of aromatic compounds. The peak at 3400 cm^{-1} showed NiO NPs. Ezhilarasi et al.[45], have prepared NiO nanoparticles using *Moringa oleifera* plant extract. Further FTIR spectra of the prepared NiO nanoparticles exhibited several characteristic bands. A broad band at 1618 cm^{-1} and 3600 cm^{-1} indicates the presence of water and hydroxyl groups on the nanoparticle surface. A sharp absorption band at 2316 cm^{-1} was observed, which is due to the stretching vibration mode of CO_2. This band may be caused by aerial CO_2 or CO_2 trapped inside the nanoparticles. This suggests that the nanoparticles rapidly adsorb water and carbon dioxide from the atmosphere, indicating a high surface area. Another broad band at 1840 cm^{-1} corresponds to the aromatic group

present in the *Moringa oleifera* plant extract and indicates the presence of aromatic compounds from the plant extract in the nanoparticles. The metal-oxygen stretching frequencies in the range of 400–1000 cm^{-1}, specifically at 965 cm^{-1}, indicate the presence of Ni-O bonding. This suggests the presence of Ni-O bonds in the nanoparticles.

3.2 *Transmission Electron Microscopy (TEM)*

Transmission Electron Microscopy (TEM) is extensively used to analyze the internal composition of synthesized NiO-NPs with respect to their morphology, particle size, and size distribution [58,59]. Synthesized MO-NPs either have good dispersion or agglomeration. The synthesized secondary nanoparticles are found to be agglomerates of various primary particles through weak der Waals or capillary forces (soft agglomerates) or strong chemical bonds (hard agglomerates). This behavior can be judged from TEM images [34,35,60–64]. The particle size can be confirmed through the plot of the number-frequency histogram particle size distribution. HRTEM is more advanced than TEM because it combines both transmitted and scattered electrons to obtain a high-resolution image [65–68]. This technique provides a clear and high-resolution image of the inner structure and hence provides information about a particular structure, unlike TEM, which provides a statistical assessment of NP morphology [69–74].

Mirza et al. [28], biomedically synthesized NiO-NPs using *Toona ciliata*, *Ficus carica*, and *Pinus roxburghii* plant extracts. They perform TEM analysis, which confirms some NiO-NPs to be irregular, cube-like, cylindrical, rod-like, and octahedral in shape. Some degree of agglomeration was also reported. The size of NiO-NPs was measured using Image J software and was found to be in the range of 10–70, 47–53, and 41–47.5 nm diameter for *Ficus carica*, *Toona ciliata*, and *Pinus roxburghii*, respectively. Morphological and structural analysis of *Moringa oleifera*-mediated synthesis of NiO-NPs was performed using HR-TEM. This analysis shows a small size, spherical shape, and slight agglomeration in green synthesized NiO-NPs [45].

3.3 *Scanning Electron Microscopy (SEM)*

Scanning electron microscopy reveals the chemical composition of nanoparticles and surface morphology by using a high-energy electron beam produced by a heat filament to scan the surfaces [75–77]. Mirza et al. [28], synthesized NiO-NPs using plant extracts of *Toona ciliata*, *Ficus carica*, and *Pinus roxburghii*. Further, the surface morphology was studied using Scanning electron microscopy (SEM), and the results show the aggregation of smooth solids of different shapes and sizes in *Toona ciliata* plant extract, brick-shaped NiO-NPs in *Pinus roxburghii* plant extract, and granular shapes in *Ficus carica* plant extract. NiO nanoparticles surface morphological study was carried out using Field emission scanning electron microscopy (FESEM) by Ali et al., and the images obtained clearly show spherical structure and a little agglomeration in NiO-NPs. Four peaks of the Ni element were found in the Energy Dispersive X-ray Spectroscopy (EDS) [50]. The Scanning electron microscopy (SEM) analysis of *Vernonia amygdalina* (Leaves)-mediated NiO-NPs carried out by Asratemedh in *Moringa oleifera* confirmed octahedral structure with cubic face-centered geometry and matches with bunsenite phase [62]. Motene et al. [36], synthesized a flower-like NiO nanoparticle by using *Sutherlendia frutescene* extract. The morphological and elemental analysis by using Field emission scanning electron microscopy (FESEM) and Energy Dispersive X-ray Spectroscopy (EDS) techniques shows radially growing cauliflower-like structures. A possible reason for the formation of a flower-like shape could be hydrolysis (in the presence of polyphenol). Ni-OH nanosheets, due to their flexible, wrinkled, and bent properties, form clusters with each other, resulting in a flowerlike shape. The morphological analysis of NiO-NPs synthesized from *Salvia hispanica* L. (chia) seeds was studied using the Field emission scanning electron microscopy (FESEM) technique, and their elemental combination was confirmed using the Energy Dispersive X-ray Spectroscopy (EDS) technique, which shows peaks of Ni and O [37].

3.4 *X-ray Diffraction (XRD)*

X-ray Diffraction, on account of its non-destructibility, is a widely used technique to determine the crystallinity of the sample. Researchers have evaluated XRD patterns for studying phase identification of some plant extracts with 2θ geometry and their diffraction peaks, as given in Table 2.

Table 2. XRD patterns of various plant extracts

Sr. No.	Plant Extracts	Diffraction Peaks	MO-NPs/NPs	Diameter (nm) and Identification	Reference
1.	*Toona ciliate* *Ficus carica* *Pinus roxburghii*	37.33° (111), 43.28° (200), 62.91° (220), 75.50° (311), 79.51° (222) 37.32° (111), 43.54° (200), 62.91° (220), 75.49° (311), 79.36° (222) 37.48° (111), 43.56° (200), 63.06° (220), 75.64° (311), 79.51° (222)	NiO NiO NiO	11.86, FCC 16.43, FCC 17.47, FCC	28
2.	*Opuntia ficus indica*	37.14° (111), 43.35° (200), 62.89° (220), 75.49° (311), 79.26° (222)	NiO	16.01, Cubic	38
3.	*Cocos nucifera*	38.2°, 44.4°, 77.7°	NiO	-, FCC	62
4.	Egg white	31.19° (111), 45.13° (200), 56.29° (220), 72.64° (311), 75.05° (113)	NiO	30, -	55
5.	*S. aureus*	37.10° (111), 43.32° (200), 62.81° (220), 76.51° (311)	NiO	32.9, Hexagonal & FCC	56
6.	*Lactuca serriola* (Seeds)	44.4° (111), 52° (200), 76.4° (220)	Ni	Mixed phase structure	57
		37.1° (111), 43.1° (200), 62.7° (220), 75.1° (311)	NiO		
7.	*Vernonia amygdalina* (Leaves)	35.6° (111), 37.22° (222), 43.24° (400)	NiO	17.86, Cubic	61
8.	*Sutherlendia frutescence* (leaves)	38.6° (111), 44.1° (200), 63.1° (220), 76.3° (311), 80° (222)	NiO	18.4, FCC	62
9.	*Moringa oleifera* (Leaves)	37.25° (111), 43.26° (200), 63.11° (220), 75.46° (311), 79.45° (222)	NiO	9.69, -	45

3.5 *UV-visible spectroscopy*

UV-visible spectroscopy is an extensively used non-destructive technique concerning UV (185–400 nm) and visible (400–700 nm) light. V. Haritha et al.[7], have synthesized NiO-NPs by using *Averrhoa bilimbi* fruits. UV-Visible analysis shows maximum absorption at 340 nm, which confirms the presence of NiO. The electron excites from the valence band to the conduction band, i.e., from the 2p orbital of O to the 3d orbital of Ni. Due to the following electron transfer from ligand to metal, a significant absorption peak is observed. Lingaraju et al.[12], have used the UV-DRS technique to record the spectra of biosynthesized NiO-NPs in the range of 300–800 nm. Now, due to the same reason discussed above, i.e., the electron excitation from the valence band to the conduction band, the absorption peak is observed at 321 nm. Olajire et al.[15], also synthesized NiO-NPs using leaf extracts of *Ananas comosus* and characterized them by UV-visible spectroscopy. It has recorded the spectrum at λ max 203 nm (K-band) and 206 and 208 nm (band II). Due to the presence of the benzoyl system, the K band has a π-π* transition, while wavelengths of 260 and 208 nm indicate either a combination of π-π* and n-π* transitions or n-π* transitions, which confirms a double bond linked with heteroatoms. Some other peaks at 315, 349, 365, and 700 nm were seen in the spectrum of Ni (II) ions, which were absent in the NiO spectrum. This directs the formation of NiO-NPs from Ni ions during the bio-reduction process.

3.6 *Energy Dispersive X-ray Spectroscopy (EDS)*

Energy Dispersive X-ray Spectroscopy (EDS) analysis is a micro-analysis technique used for determining the purity percentage and elemental composition of biogenic NiO-NPs. Different elements in the sample have distinct spectral peaks, and by studying them, elements associated with nanoparticles can be identified. It is a non-destructive technique that has the ability to perform microscopic imaging by slightly modifying it with SEM or TEM instruments[43,78–81]. Ezhilarasi et al.[45], synthesized NiO-NPs from *Moringa oleifera*. Elemental analysis of small-sized and spherical-structured metal oxide NPs was done using EDS, which approves the formation of pure Ni and O elements. Ni and O peaks were clearly seen in the EDS spectrum. Mirza et al.[28] synthesized NiO-NPs from *Toona ciliate*, *Pinus roxburghii*, and *Ficus carica* and characterized them by UV-visible spectroscopic, FTIR, XRD, SEM, TEM, and elemental analysis by using energy dispersive X-ray spectroscopy. The weight percentage of NiO-NPs derived from *Toona ciliate*, *Pinus roxburghii*, and *Ficus carica* was determined. In *Toona ciliate* plant extract, elements like N (6.25%), O (51.53%), Na (18.99%), and Ni

(23.23%) were found. In *Pinus roxburghii*, N (9.63%), O (48.72%), Na (17.39%), and Ni (24.26%) were reported, while N (8.82%), O (50.35%), Na (14.32%), and Ni (26.51%) were reported in *Ficus carica*[28].

4. Applications of Ni/NiO-NPs

Researchers have developed nickel and nickel oxide nanoparticles by various methods and evaluated their biological applications like antibacterial, antioxidant, anticancer, anti-inflammatory, anti-larvicidal, antifungal, anti-diabetic activities, and photocatalytic degradation of dyes. Reported biological applications and photocatalytic degradation of dyes have been discussed thoroughly.

4.1 *Biological applications of NiO and Ni nanoparticle*

4.1.1 *Antibacterial activity*

The antibacterial activity of NiO nanoparticles is evaluated against various strains of gram-negative bacteria (*P. aeruginosa*, *E. coli*) and gram-positive bacteria (*B. subtilis*, *S. aureus*) by using the agar well diffusion method and the agar disc diffusion method, as shown in Figure 6. In the agar-well diffusion method, McFarland standards are prepared on an agar plate. Further, with the help of 8-mm sterile cork-borer wells on agar plates. NiO-NPs solutions of different concentrations are poured into each well and incubated at 37 °C for 24 h. After incubation, the zone of inhibition (a clear zone diameter around each well) formed indicates inhibition of bacterial growth and is measured in mm[82,83]. In the agar disc diffusion method, nutritional agar medium, or Muller-Hinton agar medium, is prepared, allowed to solidify, and sterilized in an autoclave for 20 min. The gram-negative bacteria (*E. coli*) and gram-positive bacteria (*S. aureus*) were dispersed over the prepared solidified media to obtain the bacteria's strain. The sterile filter paper containing samples of different concentrations of NiO-NPs was prepared and spread on the bacterial lawn. They were incubated for 24 h at 37 °C. The inhibitory zone was seen around the filter paper containing different concentrations of NiO-NPs, which indicates the action of NiO-NPs against bacterial strains. In both methods, antibiotics such as amikacin, cefopera zone-sulbactam, cefotaxime, gentamicin, tobramycin, piperacillin, piperacillin-tazobactam, amoxycillin, co-trimoxazole, levofloxacin, imipenem, cefepime, sparfloxacin, cefopera zone, ciprofloxa, cinampicillin, kanamycin, erythromycin, and chloramphenicol were used as standards, and their antimicrobial activity was compared with the activity of synthesized NiO-NPs[84–86]. Antibacterial activities of NiO NPs synthesized using various plant extracts against the bacterial strains are discussed in Table 3.

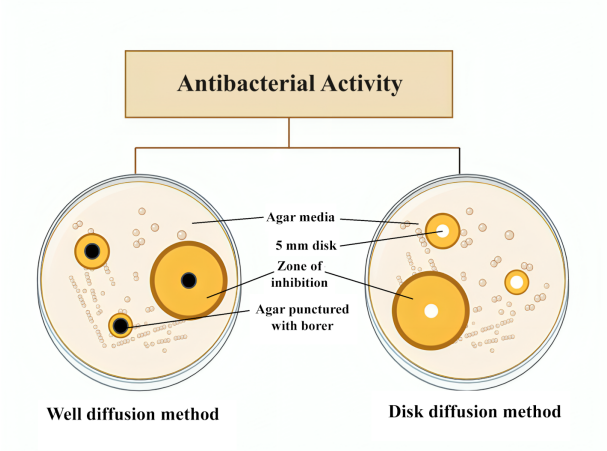

Figure 6. Methods of determination of antibacterial activity

Table 3. Antibacterial activities of biosynthesized NiO-NPs

Plant Extract	Agar Medium	Bacterial Strains	Maximum Zone of Inhibition (mm)	NiO NPs Conc. (µg/mL)	Reference
Berberis balochistanica	Muller Hinton	*P. vulgaris* *S. aureus*	16 18	1000 µg/mL 1000 µg/mL	2
Rhamnus virgata	Sabouraud dextrose	**Gram-negative** *E. coli* *P. aeruginosa* *K. pneumoniae* **Gram-positive** *S. aureus* *B. subtilis*	 26 16 14 18 28	 1000 µg/mL 1000 µg/mL 1000 µg/mL 1000 µg/mL 1000 µg/mL	3
Raphanus sativus	Nutrient agar	*B. subtilis* *S. aureus* *E. coli* *P. aeruginosa*	2.176 2.189 4.175 2.576	-	16
Averrhoa bilimbi	Nutrient agar	**Gram-positive** *S. aureus* **Gram-negative** *E. coli*	 6.10 7	 150 µg/mL	43
Euphorbia heterophylla	Nutrient agar	**Gram-positive** *S. aureus* **Gram-negative** *E. coli* *P.desmolyticum* *K. aerogenes*	 8.23 9.23 7.33 7.10	 600 µg/mL 600 µg/mL 600 µg/mL 600 µg/mL	44
Camellia sinensis (Leaf)	Nutrient agar	*P. aeruginosa* *S. epidermidis*	23.13 ± 0.15 12.00 ± 0.216	100 µL	24

4.1.1.1 *Mechanism of antibacterial activity*

Metal oxide NPs, when treated with bacteria, break the growth or multiplication of the colony, and this activity is known as the bactericidal effect or bacteriostatic effect of MO-NPs. Although the exact mechanism of bacterial cell death is undefined, some probable mechanisms are discussed. In one of the mechanisms, liberation of antibacterial metal ions from MO-NPs is expected. According to the mechanism, MO-NPs, when dissolved in water containing bacteria, liberate antibacterial metal ions and thus increase the bactericidal effect. The action of the antibacterial metal ion on the envelope of the bacterial cell is explained by other possible mechanisms where functional groups like thiol (−SH), amino (−NH), and carboxylic (−COOH) groups of proteins present in the cell wall of bacteria react with the antibacterial metal ion, leading to their denaturation. The bacterial cell death due to the action of antibacterial metal ions includes degradation of the lipopolysaccharide and denaturation of proteins, destruction of lipids in cell membranes, separation of the breathing process due to the attachment of metal ions to respiratory chain proteins, and protein inhibition present in the cytoplasm and ribosomes, which eventually leads to cell death. In another well-known mechanism, reactive oxygen species (ROS) are formed on the nanoparticle surface, leading to oxidative stress generation. Vital cell components like proteins, polyunsaturated fatty acids, and nucleic acids are attacked by ROS generated on the surface of nanoparticles [87–90].

4.1.2 *Antifungal activity*

Metal Oxide Nanoparticles possess antifungal activity, and their fungicidal mechanism has greater potential than commercial drugs. Moreover, commercial drugs have their own disadvantages, like limited clinical applications and adverse side effects like liver damage, renal failure, nausea, diarrhea, and an increase in body temperature. To overcome this problem, biosynthesized metal oxide NPs are the best solution. It breaks the membrane and thus damages the fungal intracellular component, destroying cell functions. The antifungal activity of NiO-NPs is checked against various fungal pathogens. In the literature, various researchers have studied them. Iqbal et al. [3], synthesized NiO-NPs using *Rhamnus virgata*, and their antifungal activity was evaluated against different fungal strains such as *Aspergillus flavus*, *Candida albicans*, *Mucor racemosus*, *Aspergillus niger*, and *Fusarium solani*. The fungal strains were cultured using Sabouraud dextrose agar media on media plates. Filter discs loaded with NiO-NPs sample solutions having different concentrations (700–10.9375 µg/mL) were allowed to spread on the fungal-infected media lawn. Amphotericin B was used as a positive

control, while DMSO was used as a negative control. The zone of inhibition was measured by a Vernier caliper after the incubation period[18].

Uddin et al.[2], reported that *Berberis balochistanica* leaves extract are used for synthesis of NiO-NPs act they act as capping, reducing and stabilizing agents. Further they examined their antifungal activity using poisoned food method. Antifungal activity of NiO-NPs was tested against different Phytopathogenic fungal pathogens such as *Asperagalusniger*, *Alternaria alternate*, and *Fusarium oxysporum*, *Penicillium* spp. SDA (Sabouraud Dextrose Agar) medium was prepared and autoclaved. NiO-NPs of different concentrations (100, 500, and 1000 µg/mL) were mixed with SDA medium, and filter discs (5 mm) loaded with 7-day-old culture were placed on the prepared SDA medium. Growth was measured in mm after the inoculation period[24].

4.1.3 *Anticancer activity*

Cancer is a disturbing disease and one of the main causes of the increase in mortality rates worldwide. Though cancer is a treatable disease, its different therapies have side effects, as it is difficult to differentiate between normal and cancerous cells. Nanomaterials were first studied in 1970 for carrying antitumor pharmaceuticals by entrapping in liposomes. Iqbal et al.[22], have analyzed the anticancer activity of NiO-NPs, which has been determined by the MTT [3-(4,5-dimethyl thiazol-2yl)-2,5-diphenyl tetrazolium bromide] assay against human liver cancer cell line (HepG2) cells. In the MTT assay, tetrazolium salt is reduced by mitochondrial dehydrogenase in viable cells. The RPMI 1640 medium, added with 10% fetal bovine serum, was used to culture HepG2 cells and was maintained at 37 °C under 5% CO_2 in a humidified atmosphere. Further, the method of trypsinization, i.e., 0.025% trypsin and 0.0025% EDTA, was used to subculture cells, which were maintained in a tissue culture laboratory. For growing cells, 96-well microtitre plates were used at 1.25×10^4 cells/well for 24 h. These cells were grown for the MTT assay. Once this procedure was done, the full-fledged cells were washed twice with 100 µL of serum-free medium, starved for an hour at 37 °C in a CO_2 incubator, and treated with different concentrations of NiO-NPs (500–3.9 µg/mL) for 48 h. Furthermore, MTT reagent (10%) was added to each well and allowed to incubate in a water bath at 37 °C for 4 h. DMSO (10%) was also added, and the plates were shaken in the dark using a plate shaker. Finally, the absorbance was measured at 590 nm for each sample in order to calculate the anticancer potential of NiO-NPs. The results of the respective MTT assay were expressed as the IC50 values (effective concentration showing 50% inhibition of activity)[26,27]. The results show the highest inhibition with ~84% mortality at 500 µg/mL in dose-dependent inhibition. The anticancer activity decreases with a decrease in concentration. The IC50 value of 39.7 µg/mL was calculated, which indicates that the sample was in the category of general cytotoxic[7].

4.1.3.1 *Mechanism of anticancer activity*

MO-NPs inhibit cancerous cells, and their exact mechanism is not known but the probable mechanism is discussed as shown in Figure 7. It is suggested that the NiO-NPs enter the cell and induce intracellular oxidative stress, which disturbs the stability between oxidants and antioxidants. The oxidative stress of microbial cells is intensified by ROS formation on the surface of NPs. In this oxidative stress situation, the mitochondrial electron transport chain forms the anticancer mechanism of ROS. Now, to reduce damage caused to the cell by ROS, free radicals get converted into less toxic molecules via enzymes. For example, hydrogen peroxide is formed by superoxide dismutase (SOD), which in turn is converted to H_2O (a less toxic molecule) in the presence of glutathion peroxidase enzymes. Due to this reaction, there is an imbalance in cells, which leads to cell death. Haritha et al., reported that the cell population of cancer cells (HCT 116 and L929) was decreasing with an increase in NiO-NPs solution concentration (20 µg/mL to 100 µg/mL). This is due to the fact that the Ni^{2+} was interacting with the cancer cell membrane and causing injury to it[7,91–93].

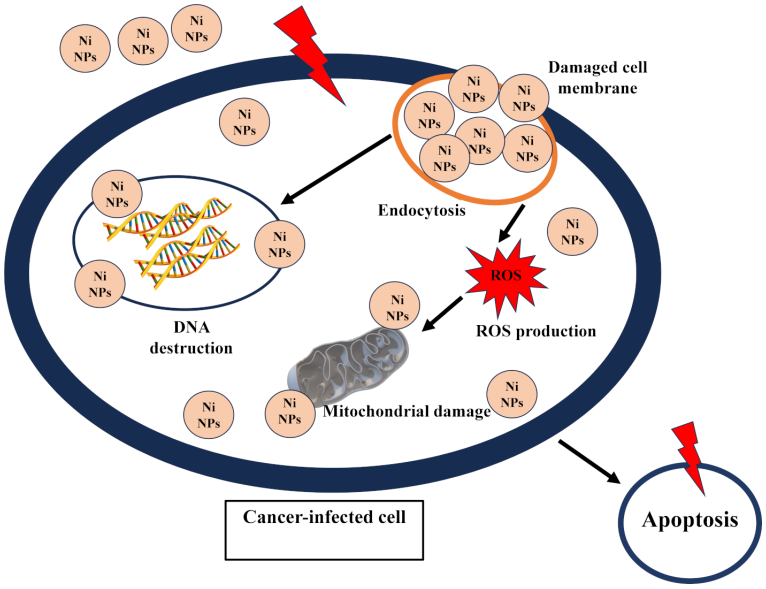

Figure 7. Mechanism of anticancer activity

4.1.4 *Antidiabetic activity*

Diabetes mellitus is a complex endocrine disorder that affects carbohydrate, fat, and protein breakdown. About 10% of the worldwide population is affected by diabetes mellitus. Low insulin secretion affects the health of multiple organs and thus increases the risk of cardiovascular diseases (CVD), peripheral vascular diseases (PVD), stroke, neuropathy, renal failure, retinopathy amputations, and blindness. The blood sugar level of the human body is controlled by pancreatic endocrine hormones, i.e., insulin and glucagon. The β-cells present in the islets of Langerhans secrete insulin, which potentiates the ability to absorb glucose out of the blood by muscle, red blood cells, and fat cells. The absorbed glucose is used in other metabolic processes, thus maintaining the sugar level of the body. While glucagon is secreted by α-cells of the pancreas, which stimulates the liver and other cells to release glucose out of the stored block in our bodies, For the treatment of diabetes, insulin and various types of hypoglycemic agents, such as biguanides and sulfonylureas, are used. But these medications, after prolonged use, come with several harmful and toxic side effects. To overcome this problem, Ni and NiO nanoparticles are used, which show potential anti-diabetic properties. The anti-diabetic properties of NiO-NPs are analyzed using the α-amylase inhibition method[94,95].

Shwetha et al.[95], has prepared 1 mL of phosphate buffered saline (PBS) solution and 0.5 mL of Ni-NPs solution of different concentrations (100, 200, 300, 400, and 500 µg/mL). Further 200 µL of 0.5 mg/mL α-amylase and 200 µL of 5 mg/mL starch solution were added, followed by 10 min of incubation at room temperature. Starch with α-amylase and without α-amylase was taken as control. To quench the reaction, 400 µL of Dextrose Normal Saline (DNS) solution was added. Finally, the reaction mixture was heated in a boiling water bath for 5 min and then cooled down. The metformin drug was used as a standard, and enzyme activity inhibition was calculated using a standard formula.

$$\% \text{ Inhibition of Enzyme Activity } = \frac{\text{Abs Sample } - \text{ Abs Control}}{\text{Abs Sample } \times 100} \quad (1)$$

It has been observed that an IC_{50} value of 232.12 µg/mL indicates the inhibitory effect of the standard drug Metformin on α-amylase activity, while an IC_{50} value of 268.13 µg/mL indicates the inhibitory effect of synthesized NiO-NPs on α-amylase activity[95]. Hongying Gao et al.[96], studied the anti-diabetic properties of Vitex pseudo-negundo leaf extract (VPLE)-mediated NiO-NPs on the livers of streptozotocin-induced diabetic rats. The liver-protecting effects of VPLE in an STZ-induced diabetic hepatopathy model were examined, and results showed improvements and regulated levels of FBS,

ALT, AST, ALP, and Alb in diabetic rats compared to untreated diabetic rats. With the increase in VPLE concentration level, the effectiveness also increased (500 mg kg^{-1} VPLE has more effectiveness than 250 mg kg^{-1} VPLE).

Haritha et al.[7], studied α-Amylase inhibition of synthesized NiO-NPs. One of the important enzymes for diabetic management is considered to be pancreatic α-amylase (class, α-1,4-gluconohydrolases). The IC_{50} value of prepared NiO-NPs was found to be significantly greater than the value of standard metformin. *A. bilimbi* fruit-mediated NiO-NPs has an IC_{50} value of 311.26 µg mL^{-1}, while metformin has a value of 307.14 µg mL^{-1}. With the increase in concentration of NiO-NPs, inhibition increases gradually.

4.1.5 *Anti-larvicidal activity*

In previous scientific studies, the anti-larvicidal properties of Ni-NPs were studied thoroughly. Most of the developing countries are facing health threats due to mosquito-borne illness. The mosquito bites human beings, and it is responsible for causing diseases such as filariasis, dengue fever, malaria, and epidemic encephalitis[97]. Elango et al., synthesized Ni nanoparticles by using *C. nucifera coir* methanolic extract and characterized their surface, structural, elemental, and morphological properties by using standard characterization techniques like UV-visible, XRD, FT-IR, SEM, TEM, Zeta potential, and GC-MS. Further anti-larvicidal assays of synthesized Ni-NPs were performed against larvae of a dengue-causing vector known as *Aedes ageypti*. The activity was performed by placing 20 dengue-causing larvae (*Aedes ageypti*) in distilled water and Ni-NPs solutions of various concentrations (100–500 ppm). Larvae placed in plain distilled water were considered controls. After the exposure of Ni-NPs to larvae for 24 h, the mortality rate was evaluated. Also, ANOVA, LSD, and Tukey's test were used for calculating LC_{50} and LC_{90}. The mortality rate varied with the change in concentration discussed in Table 4. The LC_{50} and LC_{90} values were calculated to be 259.24 ppm with the LCL of 224.64, the UCL of 294.18 ppm, and 446.99 ppm with the LCL and UCL of 398.44 and 520.17, respectively. ANOVA LSD Tukey's test was used for calculating the chi square value to be 6.869[98].

Table 4. Anti-larvicidal assay of synthesized Ni-NPs at different concentrations

Vector	Concentration (ppm)	Mortality Rate (%)
Aedes ageypti	100	18.4 ± 1.2
	200	36.6 ± 1.8
	300	58.8 ± 2.2
	400	78.6 ± 2.8
	500	97.5 ± 2.6

Prabhu et al.[99], evaluated the anti-larvicidal activity of Ni-metal organic frameworks (MOFs) against the third instar stage *A. aegypti* mosquito larvae. The mortality rate was observed to be maximum at 75 µg/mL, and LC_{50} and LC_{90} values were found to be 43.62 ± 1.25 and 74.72 ± 1.97 µg/mL, respectively, against *A. aegypti* mosquito larvae. The mortality rate increases with a rise in the concentration of Ni-MOFs. The nano-sized NiO-MOFs penetrate the midgut epithelial cell and thus retard the life of larvae. Cell cycle arrest and a decrease in growth rate might be the main causes of the apoptotic effect of nickel, which leads to mosquito larvae death. Varied Ni-MOFs concentrations affect the action against mosquito larvae. Low concentrations cause growth retardation, while high concentrations cause organelle damage such as head, siphon, and thorax damage.

Adequate information about its drug delivery system, antioxidant properties, catalytic activities, dye adsorption from industrial waste, dye sensitized solar cells and sensors have been added in the Supplementary Material part.

5. Conclusions

Plant-mediated and microorganism-based synthesis are the most accepted and advanced methods for the green synthesis of nickel and nickel oxide nanoparticles. Phytochemicals such as alkaloids, flavonoids, terpenoids, polysaccharides, and heterocyclics have been used as suitable capping agents. These methods are preferred over chemical and physical methods

due to their eco-friendly, economic, and non-toxic nature. Plausible mechanisms of interaction between phytoconstituents and metal ions are not well distinguished. Various characterization techniques, such as FT-IR, SEM, TEM, UV-Visible, XRD, and EDS, are adopted for studying the structural and morphological parameters of Ni/NiO-NPs. Hence, more research on the mechanistic details should be meted out for dipper insights in the synthesis of Ni/NiO-NPs. This paper provides glimpses of potential enhanced activities such as antifungal, antibacterial, anti-larvicidal, anticancer, antidiabetic, antioxidant activities. and drug delivery systems of biogenic synthesized Ni/NiO-NPs. Ni/NiO-NPs, due to their particle size distribution, surface area, volume, and shape, have potential applications such as photocatalytic activity, dye sensitized solar cells and sensors, dye adsorption from industrial waste, and catalytic activities. Mechanistic approaches to interaction between synthesized Ni/NiO-NPs and various microbes are discussed, and low cytotoxicity is the major drawback of these nanoparticles. These characteristics can be effectively implemented in various fields of sustainable development and environmental remediation.

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Confilict of interest

The authors declare no competing financial interest.

Supplementary Materials

Table S1. Table of contents

Ni/NiO-NPs in drug delivery system

A nanoparticle-based drug delivery system is an advanced engineered technique for prolonging the systemic circulation, sustainable release, preferential targeting, and releasing drugs in a controlled manner. It has been reported that nanoparticles loaded with drugs through endocytosis enter the host cells and release the drug to treat various infections. The main objective of the drug delivery system is to maximize effect healing activity and minimize the adverse effect of drugs. The nanostructured materials used in drug delivery systems are classified as metallic NPs, carbon-based NPs, semiconductor NPs, polymer-based NPs, and nanocomposites. It has proved that metallic NPs have prominent therapeutic applications. Characteristics of particles, such as particle size distribution, surface area, volume, and shape, are related to the effectiveness of the drug delivery systems. Owing to the small size and large surface area of Ni/NiO-NPs, they hence show enhanced drug solubility and dissolution [100,101]. Adhikary et al. [102], have delivered erythromycin against erythromycin-resistant *E. coli* and *S. aureus* by conjugating erythromycin on the surface of a fabricated NiO-NPs-based drug delivery system to develop NiO(I)-Ery, NiO(Br)-Ery, and NiO(Cl)-Ery. For the purpose, three simple mononuclear Ni(II) Schiff-base complexes were used to synthesize diversities of NiO-NPs such as NiO(I), NiO(Br), and NiO(Cl) using the pyrolytic

technique. Results show effective antimicrobial activity against Erythromycin-resistant *S. aureus* and *E. coli*. Yan Lu et al.[103], performed green synthesis to prepare NiO-NPs using *Cressa nudicaulis* plant extract and further loaded it with the doxepin drug. An effective medicine was tested against head and neck cancer as well as depression.

Antioxidant properties of Ni/NiO-NPs

Antioxidants are the stable molecules that can donate an electron to a free radical, making them stable and less reactive, as shown in Figure S1. Free radicals are unstable molecules or an ion with unpaired electrons, produced by oxidation in the body, active enough to damage the cell membrane and cause lethal heart diseases, certain cancers, and Alzheimer's. An unstable free radical, desperate to attain stability, easily reacts with other molecules, making a chain of unstable molecules. Thus, by breaking a bond of stable molecules, free radicals are formed. There are three types of free radicals on the basis of positive, negative, or neutral charge on them. They are formed in the body when unstable chemicals are produced by oxidation or by environmental factors like air pollution, tobacco smoke, or exercise. In the human body, oxygen as a free radical damages DNA and other molecules by forming ROS, which in turn cause oxidative stress. It weakens the antioxidant defense mechanism and is considered a biological attack on the body. It causes damage to the body, which includes premature aging, diabetes, cancer, eye damage, heart failure, brain damage, muscle problems, and a weak immune system. As discussed above, antioxidants without getting destabilized donate electrons to free radicals, eventually making them less reactive and stable[104,105].

Rehman et al.[106], have used *Bergenia ciliate* leaf extract for the synthesis of NiO-NPs. Further antioxidant activity of synthesized NiO-NPs was studied against the $ABTS^{\bullet+}$. Results show that antioxidant activity increases with an increase in the concentration of the sample. It suggests that to neutralize $ABTS^{\bullet+}$ cationic radical, a larger number of antioxidant molecules are required. Yu Zhang et al.[107], synthesized NiO-NPs using aqueous extract from the leaves of *Calendula officinalis,* and the antioxidant activity of the synthesized NiO-NPs was investigated using the DPPH test. The sample at a concentration of 204 µg/mL prevented 50% oxidation of the DPPH molecules. Firisa et al.[108], synthesized NiO-NPs and Cu-NiO nanocomposite using the green method (leaves extract of *Phytolacca dodecandra* L'Herit). Further, they evaluated the antioxidant activities of synthesized nanoparticles. The result shows that NiO-NPs and Cu-NiO nanocomposite inhibited oxidation of 50% of the H_2O_2 molecules at concentrations of 363.96 and 350.29 µg/mL respectively.

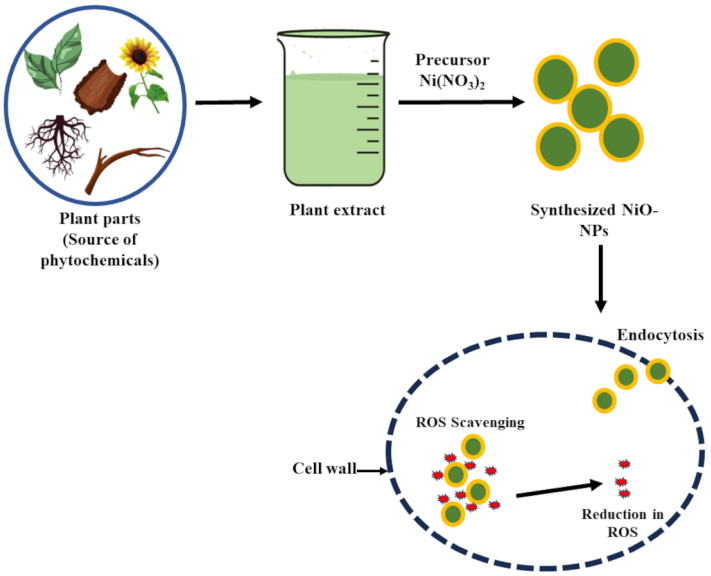

Figure S1. Antioxidant mechanism of NiO-NPs

Catalytic activities

Catalysis is the activity of the substance that enhances the rate of reaction without getting consumed, as shown in Figure S2. There are two types of catalysis, i.e., homogeneous and heterogeneous. Catalysis using metal nanoparticles is considered semi-heterogeneous catalysis. Determination of the true nature of the Ni-NPs as the true catalyst was demonstrated using TEM, kinetic, poisoning, and filtration experiments. Further, they successfully used Ni-NPs as a catalyst in the hydrogen-transfer reduction of carbonyl compounds [109]. Nickel nanoparticles have potential applications as catalysts due to their low cost and availability. In cross-coupling reactions, nickel nanoparticles can be used as a catalyst. In the oxidative addition process, the cleavage of aryl ether is possible using the given catalyst [110,111]. Ni/NiO-NPs, due to their strong magnetic and interfacial properties, have catalytic applications in emulsion separation, purification of water, cleaning of oil spills, and remediation of impure samples. Chaudhary et al. [112], studied application of Ni-NPs as a catalyst in Knoevenagel condensation reactions of aromatic aldehydes and malononitrile under solvent-free conditions and also checked the reusability of the catalyst. Manna et al. [113], prepared Ni-NPs supported on cobalt ferrite ($Ni^0/CoFe_2O_4$), polydopamine-coated cobalt ferrite (Ni^0/PDA-$CoFe_2O_4$) or silica-coated cobalt ferrite (Ni^0/SiO_2-$CoFe_2O_4$). Further, they studied their catalytic properties for the hydrolysis of ammonia borane to generate hydrogen. Jiang et al. [114], chemically synthesized Ni-NPs and studied their catalytic activity. It showed excellent catalytic activity in the reduction of p-nitrophenol compared to the commercial Raney Ni. Gong et al. [115], successfully perform hydrogen evolution reactions using Ni-NPs as a catalyst under alkaline conditions and reported to be comparable in activity with most of the acid-based hydrogen evolution reactions and platinum-based catalyst reactions. Ragupathi et al. [116], reported selective oxidation of benzyl alcohol to benzaldehyde by using the highly effective optoelectronic property route of Ni-NPs.

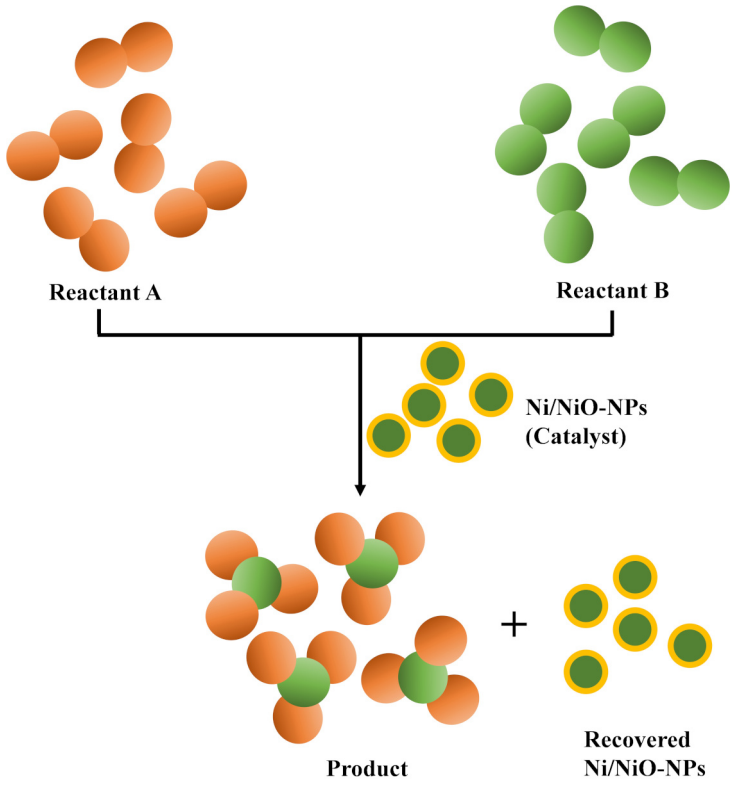

Figure S2. Illustration of Ni/NiO-NPs as a catalyst

Dye adsorption from industrial waste

Organic dyes through industrial effluent have direct hazardous effects on ecosystems. Water quality is day by day depleting, causing a lethal impact on the health of freshwater. These harmful dyes enter the ecosystem through a water source, hence causing the biomagnification and thereby impacting human health. NiO-NPs showed high adsorption rates when used for the adsorption of a Congo red dye. Adsorption isotherm and kinetic studies reveal the high effectiveness of NiO-NPs against the organic dyes. Adsorption of anionic dyes such as methyl orange, methyl blue, and Congo red onto NiO-NPs was performed effectively [117–119]. Hatem A. Al-Aoh et al. [120], studied the adsorption of bromophenol blue dye on NiO-NPs at different temperatures. The test confirms the significant adsorption ability of NiO-NPs to remediate the harmful dye from wastewater. The process of adsorption using the dye is an endothermic process according to the thermodynamic parameters such as delta ΔH^o, ΔS^o, and ΔG^o. Bani-Fwaz et al. [121], synthesized NiO and Ni/NiO by thermal decomposition of nickel acetate tetrahydrate and bis(acetylacetonato) nickel (II) complex and used it as an adsorbent for the adsorption of crystal violet dye (organic dye) from aqueous solution. They reported the highest adsorption rate of crystal violet dye onto NiO-NPs at pH 9–10.

Dye sensitized solar cells and sensors

A dye-sensitized solar cells (DSSCs) is a low-cost thin-film solar cell based on photoelectrochemical processes in which the dye absorbs photons or light from any source to get excited and further emits electrons to be oxidized into the TiO_2 photoanode electrode. In early 1991, O' Regan and Gratzel developed the dye-sensitized solar cells (DSSCs). Generally, the dye-sensitized solar cells have transparent conductive oxides, a semiconductor film (TiO_2), a sensitizer adsorbed on the surface of the semiconductor, an electrolyte, and a counter electrolyte [122]. Krishnapriya et al. [123], have incorporated Ni nanocomposites with various nanostructured TiO_2 (interconnected bead-like, spindle-shaped, square platelet-like, and porous sphere-like) to develop dye-sensitized solar cells (DSSCs). The variation in size (15–62 nm) and morphologies (triangular and hexagonal) of Ni-nanocomposites were also incorporated to study their results. They reported improvement in the rate of electron hole pair formation and short circuit current by trapping incident light. Szindler et al. [124], synthesized NiO-NPs by the sol-gel method and used them as a semiconductor layer in the photoelectrode of dye-sensitized solar cells (DSSCs). They studied the light harvesting efficiency of electrodes and claimed NiO-NPs to be an attractive alternative to TiO_2.

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