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Green synthesis of NiO nanoparticles using *Leucas Aspera* and its antibacterial activity

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ABSTRACT
Synthesis of metal oxide nanoparticles includes different chemical/physical method, but it requires a tedious procedure, expensive chemicals, additional time, high cost equipments and also challenges our environment. The present work focuses on preparing NPs eco-friendly with the help of leaf and flower extract of *Leucas Aspera*. The structural and morphological properties are examined by XRD, SEM and EDAX. The formation of highly crystalline and pure nickel oxide particles is examined from X-ray powder diffraction. SEM image exhibits NiO NPs in spherical shape and coral reef structure for leaf and flower extract. The chemical composition of the prepared samples is confirmed from EDX analysis. The antibacterial activity is studied over gram negative and gram positive bacteria. The results depict that NiO NPs synthesized using leaf extract have a good impact on gram positive bacteria.
Keywords: *green synthesis; nickel oxide; Leucas Aspera.*

1. INTRODUCTION

To reduce the use of toxic chemicals, an eco-friendly, simple and convenient method is introduced and emerged nowadays i.e., by using different parts of plants. Metal oxide nanoparticles play an important role in R & D. It is impractical to imagine a world without modern technology. The synthesis of metal oxide nanoparticles (NiO, ZnO, MnO_2, Al_2O_3, TiO_2, RuO_2, IrO_2, MoO_2, V_2O_5, etc.) receives a great interest in physics, chemistry, material science, medical, biological, mechanical and engineering science. It plays a vital role in cosmetics [1], magnetic and optical devices [2-3], panel displays, fertilizers [4], paint pigments, sensors, protective coatings, supercapacitors [5], etc.

Metal oxide nanoparticles can be prepared either by physical or chemical methods like sol-gel, solvothermal, hydrothermal techniques, etc [6-8]. But it includes tedious procedure, expensive chemicals, more time, high cost equipments and also challenge environment. To the researchers, these problems become a big challenge. Recently, preparing metal oxide nanoparticles via green synthesis obtain tremendous attention due to the usage of fewer chemicals, economically reasonable and also it reduces environmental hazards. Green synthesis includes the use of extracts from leaf, flowers, seeds, barks of a plant and also from microorganisms (algae, fungi, yeast) [9-11].

Recently, research on Nickel oxide nanoparticles receives attraction due to its high chemical stability, wide band gap and exhibit high electrical efficiency; which is extensively employed in sensors, battery electrode, designing ceramic, electrochromic films, heterogenous catalytic material, good antibacterial activity and now in the treatment of cancer cells [12-14].

Due to the side effects of modern drugs, the medicinal value of different parts of plants receives tremendous attention nowadays, which acts as antimicrobial, antifungal and anticancer agent for infectious human disease. This led the scientist to identify new medicinal drugs without side effects from different parts of herbs, trees, shrubs and even from microorganisms. *Leucas Aspera* is a common herb grown in India, whose leaves and flowers have notorious medicinal value; they are widely used in the treatment of respiratory tract disorder, painful swelling chronic skin eruption, psoriasis, chronic skin eruptions, expectorant, diaphoretic and insecticide [15-16]. The objective of the work is to prepare NiO NPs using medicinal plant Leucas Aspera leaves and flower extract and to study its structural, morphological and antibacterial activities.

2. MATERIALS AND METHODS

2.1. Preparation of Leucas Aspera leaf and flower Extract.

Fresh leaves of Leucas Aspera are collected from the local place of Coimbatore and washed with distilled water thrice to remove dust present on the surface. These leaves are sliced into small pieces and grind well-using mortar and pestle for 15 min. 20g of leaves are allowed to boil in 100 ml of distilled water. The resultant solution is filtered using Whatmann No.1 filter paper and utilized further to prepare NiO NPs. To prepare flower extract, the same procedure has to be utilized with fresh flowers.

2.2. Preparation of NiO nanoparticles using Leucas Aspera leaf and flower Extract.

To prepare NiO NPs, nickel chloride hexahydrate was used as the precursors. 10 ml of leaf extract has to be mixed with 90 ml of $NiCl_2.6H_2O$ solution drop by drop under constant stirring for 30 min. The obtained solution was stirred continuously for 1 hr and transferred to the crucible and placed in a muffle furnace under 400°C for 4 hours. Color change has been obtained in the powder and further used for characterization. The same process has been carried out to prepare NiO NPs with flower extract.

2.3. Characterization Studies.

XRD has been recorded on X'Pert PRO diffractometer in the range of 2θ=10-80° with a step size of 0.0500° using CuKα radiation (λ=0.1540 nm) to confirm the formation of nickel oxide. FT-IR spectrum has been recorded for the identification of functional groups and band. The optical properties of the prepared nanoparticles were examined using Shimadzu-2450 UV-Vis Spectrometer and photoluminescence spectra. Scanning electron microscopy has been used to study the surface morphology like the shape and size of the particles and the composition of the material was studied using EDAX analysis.

2.4. Antimicrobial Activity.

The anibacterial property of the prepared nanoparticles will be determined by study the zone of inhibition region via agar well diffusion method. The obtained particles were interacted with bacteria *(E.coli& Saureus)* at a concentration of 250, 500, 1000 µg/ml. The sterile Muller Hinton Agar is prepared and allowed to solidify. With the help of a sterile well cutter, 6mm diameter wells were punctured with uniform spacing for various concentrations for each extract. To obtain a uniform lawn of culture, the log phase culture broth was swabbed over the plate using a sterile cotton swab.

3. RESULTS

XRD pattern reveals the structural formation of the prepared material. Figure. 1 (a & b) shows the XRD spectrum of NiO nanoparticles prepared from leaf and flower extract. The graph shows the formation of pure NiO nanoparticles without any impurity with high crystalline nature. All the diffracted peaks are indexed to cubic structure and matched with earlier reports and JCPDS card no. 89-5881. Using Debye-Schrrer formula, the lattice parameter of NiO nanoparticles is calculated as 8.353 (Å) and 8.432 (Å) to leaf and flower extract added materials respectively. Compared to leaf extract, nanoparticles obtained from flower extract shows high and sharp intensity. The grain size of the NiO nanoparticles is estimated at 40.438 and 40.263 nm.

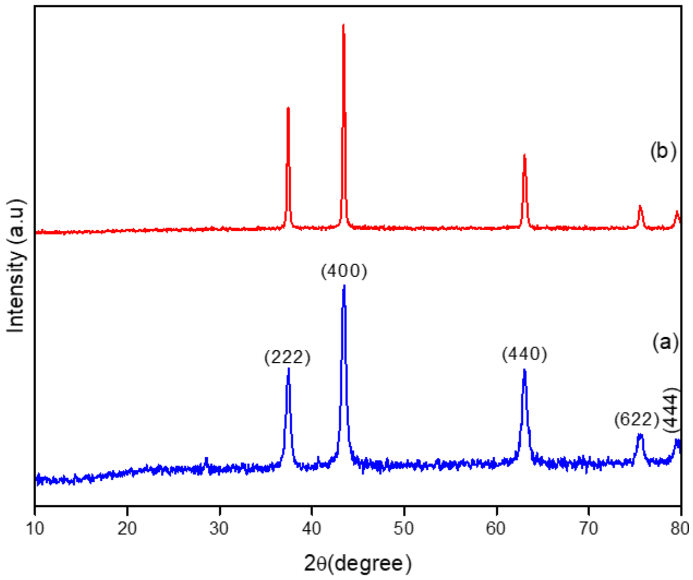

Figure 1. XRD Pattern of NiO nanoparticles using (a) leaf (b) flower extract.

Figure. 2 shows the FTIR spectrum of nickel oxide nanoparticles using Leucas Aspera leaf and flower extract in the range of 400 to 4000 cm^{-1}. The broad peak obtained at 3138 cm^{-1} attributes O-H stretching vibrations of hydroxlic groups. A peak at 1636 cm^{-1} indicates the bending mode of water molecules. The characteristics peaks at 1103 and 1403 cm^{-1} belongs to oxide groups [17].

Figure. 3 depicts the SEM images of NiO nanoparticles. Figure. 3a exhibit clear morphology with uniform distribution of spherical shaped particles in nanoscale range without any agglomeration; whereas NiO nanoparticles prepared using flower extract shows a small amount of agglomeration with coral reef structure in Figure. 3b. The chemical composition of the prepared samples was confirmed from EDX analysis. The presence of nickel and oxygen in the highly crystalline form was proved from EDX images shown in Figure. 4a. The atomic and weight percentages of the materials were obtained as 54.51, 45.49, 24.62 and 75.38 % for O and Ni respectively.

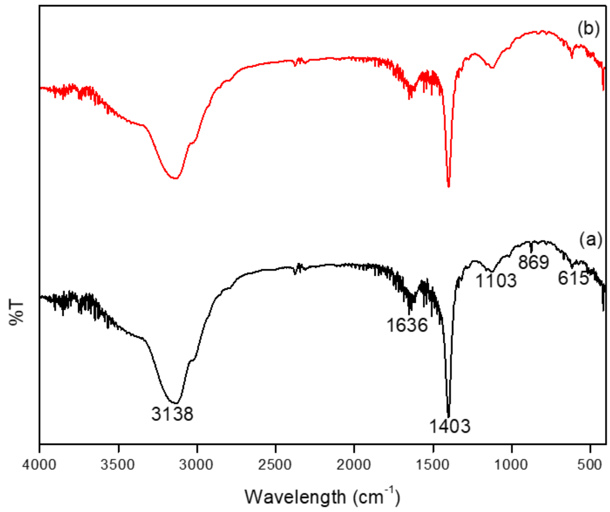

Figure 2. XRD Pattern of NiO nanoparticles using (a) leaf (b) flower extract.

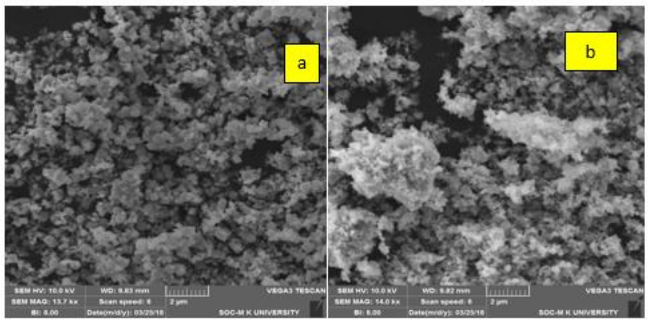

Figure 3. SEM images of NiO nanoparticles using (a) leaf (b) flower extract.

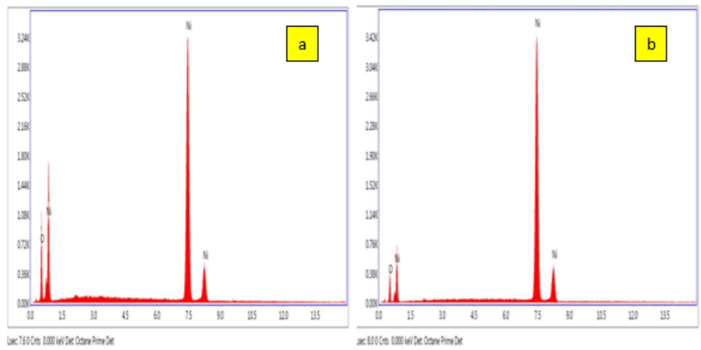

Figure 4. EDX analysis of NiO nanoparticles using (a) leaf (b) flower extract.

Antimicrobial activities depend on the particle size, powder concentration, morphology, specific surface area, etc. The zone formation helps to study the growth of bacteria and to obtain the minimum inhibitory concentration value (MIC). MIC was tested at 3 different concentrations such as 250, 500 and 1000 µg/ml over a gram positive and gram negative bacteria and the inhibition region was shown in Figure 5 and given in Table 1.

No inhibition region was observed at low concentration (250 µg/ml) for both gram negative and gram positive bacteria. By increasing the concentration, the zone of inhibition region also increases. Compared to gram negative bacteria, leaf and flower extract exhibits high inhibition region as 26 and 14 mm at 1000 µg/ml against gram positive bacteria (*E.Coli*). From the table it is also observed that the L.Aspera flower respond only at higher concentration i.e., 1000 µg/ml eventhough, less compared to the effect of leaf extract which may be due to the presence of chemicals in the leaf.

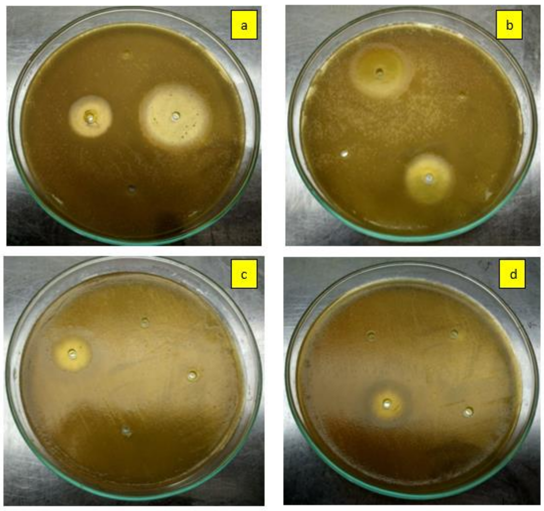

Figure 5. Antimicrobial activities of green synthesized NiO NPs (a & c) *E. Coli* (b & d) *S. Aureus* using leaf and flower extract.

Table 1. Zone of Inhibition of *E. Coli* and *S. Aures.*

Sample	Organism	Zone of Inhibition (diameter in mm)			
		1000 µg/ml	500 µg/ml	250 µg/ml	Control
L	***Staphylococcus aureus***	18	14	-	-
	Escherichia coli	26	16	-	-
F	***Staphylococcus aureus***	12	-	-	-
	Escherichia coli	14	-	-	-

4. CONCLUSIONS

The present work reports a novel, cost-effective and eco-friendly nature to prepare Nickel oxide nanoparticles using Leucas Aspera leaf and flower extract. The results elucidate the formation of highly crystalline pure Nickel Oxide particles without any impurity for both leaf and flower extract. The SEM image also indicates the formation of nanoparticles with spherical and coral reef structures. EDS analysis confirms the purity of the prepared materials. Moreover, Nickel Oxides synthesized using leaf extract shows good antimicrobial activity at 1000 µg/ml for both gram positive and gram negative bacteria.

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