Journal of New Materials for Electrochemical Systems
Vol. 26, No.2, April 2023, pp. 134-138
Journal homepage: http://iieta.org/journals/jnmes

Synthesis and Characterization of Zirconium Doped Nickel Oxide Nanoparticles Using *Acalypha indica L* Extract and Its Antimicrobial Activities

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https://doi.org/10.14447/jnmes.v26i2.a08

Received: July 19, 2022
Accepted: January 10, 2023

Keywords:
NiO Nps, Acalypha indica L, UV-Visible, SEM, FT-IR, XRD, AFM.

ABSTRACT

Nanomaterials have had one of the dimensions in the range of 1–100 nm and it's had wide range of application in various fields. The materials produces by the processing in the greener route have more sustain nature. Based on this, the investigation focused on plants based green synthesis of Zr-doped Nickel oxide nanoparticles (NiO Nps) using Acalypha indica Leaf extract for synthesis and its potential applications. The synthesized Zr doped NiO nanoparticles were characterized using Scanning electron microscopy (SEM), X-ray diffraction spectroscopy (XRD), and Fourier transform-infrared spectroscopy (FT-IR) and UV Visible spectroscopy (UV-Vis). The optical absorption calculated and confirmed by UV Visible spectroscopy, the wavelength range is 260 nm. The FCC indexing with its crystalline structure confirmed by XRD. The band at 470 confirmed the presence of NiO using FTIR, Prepared Nano sized materials present in 85 nm ensured through SEM analysis. AFM analysis of the synthesized materials represents the topography Zr doped NiO NPs deliberates good antibacterial activity.

1. INTRODUCTION

Nanoparticles are used in a broad array of applications, including medicine, semiconductors, catalysis, and energy. They are classified as particles ranging in size from 1 to 100 nm. Particles can act differently at smaller size scales than their bulk counterparts. For example, when particles get smaller, their surface area expands dramatically. This enables the emergence of features such as higher electrical and thermal conductivity, lower melting points, and greater magnetic, and unique optical qualities. The capacity to routinely use materials of this size in the creation of new materials opens up a plethora of potential in disciplines such as clean energy, catalysis, and sensors, to mention a few. Recent developments have highlighted the need of eco-friendly green chemistry technologies for the production of metal oxide nanoparticles. Plants are having chemical factories naturally. They are low-maintenance and cost-effective. Plants and plant extracts appear to be the greatest alternative for nanomaterial synthesis. [1-7]. Plants include biomolecules such as carbohydrates, proteins, and coenzymes that have a high potential for converting metal salts into metal oxide nanoparticles. The phytochemicals included in plant leaf extracts have a dual role in the nanoparticle formation process, acting as both reducing and stabilizing agents [8-12].

NiO is a p-type semiconductor metal oxide with a band gap varying from 3.6 to 4.0 eV depending on the kind and density of defects. Nanoscaled NiO particles are widely praised among all known nanoparticles due to their uses in gas sensors, solar energy, catalytic electrochemical performance, and antibacterial activities. Hereby examined the green production of Zirconium ion doped NiO NPs utilizing Acalypha indica L Leaf extract, and assessed its characteristics and prospective uses [13-17].

2. MATERIALS & METHODS

2.1 Preparation of plant extract

Acalypha indica L. leaves are esteeming in Tamil Siddha medicine, it prospective to invigorate the body easily. It having alkaloids and hydrocyanic acid and peoples in India and Africa take it as food. The plant materials collected at Sri Sarada College campus in Tirunelveli, Tamil Nadu, and India. Acalypha indica L leaves were cleaned, chopped little pieces and washed with water/deionized water several times to eliminate impurities, powdered and kept in safe. 5.0 g of powdered of materials weighed using SHIMADZU - AUX 220 weighing balance until a constant weight was obtained, then taken in 250 ml conical flask containing 100 ml of millipure distilled water. The mixture was refluxed at 90 degree for 20 minutes, cooled at room temperature, the supernatant was filtered through whatman No.1. Volume of Plant extract prepared required volume for the synthesis reaction. The aqueous extract Acalypha indica L stored at 5 degree for the further reaction.

2.2 Materials

Prepared pure plant extract, chemicals from Sigma-Aldrich viz., viz., Nickel (II) chloride ($NiCl_2{\bullet}6H_2O$) and Zirconyl

Nitrate used for the preparation. Deionized water used for the whole preparations. Streptococcus oralis (MTCC No. 2696), Aspergillus fumigatus, and Bacteriodes fragilis (Himedia (Lot- 320- 204-3) were obtained from MTCC, India. Himedia, India, supplied the Nutrient Agar medium, Nutrient broth, Gentamicin antibiotic solution, Potato dextrose agar medium, and Amphotericin B antimycotic solution. Borosil, India provided the test samples, petri-plates, test tubes, beakers, and conical flasks.

2.3 Measurements

Zr doped NiO NPs were tested by XRD using a Gonio radius 240 with Cu (λ=1.54060 Ǻ) in the range of 10 and 80. FE-SEM examination done through a high performance TESCAN MIRA3. The Atomic force microscopy analysis using the Nano surf easy 2 scan BT02218 is profilometer – a sharp cantilever tip interacts with the sample surface sensing the local forces between the molecules of the tip and sample surface. Ultraviolet Visible spectral analysis was performed on a JASCO, V-600 Diffuse Reflectance spectrophotometer. FTIR measurements of samples prepared as KBr disks were performed on a Thermo Scientific Nicolet iS5 FTIR spectrometer.

2.4 Methods

100 ml of Acalypha indica L. aqueous extract, 3.0g Nickel (II) chloride and 1.0 g Zirconyl nitrate were added in 250 ml conical flask. The solution was uniformly distributed mixed for 60 minutes with a constant stirring at 400 rpm. Observed the color of the solution changes to yellow. Then solution centrifuged, the precipitate dried in at 300°C in an oven. Dried powdered product materials sent for examination and follows in application studies [18].

3. RESULTS AND DISCUSSION

3.1 UV-visible spectra analysis

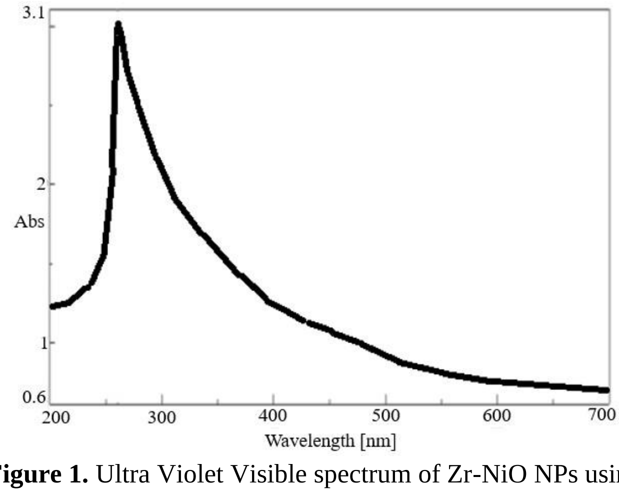

Figure 1. Ultra Violet Visible spectrum of Zr-NiO NPs using Acalypha indltica L.

Zr - NiO nanoparticles measured using Ultra Violet Visible absorption spectrum, which provides information about the molecule's structure since UV and visible light absorption involves the promotion of electrons in the orbitals from the ground state to higher states. The absorption spectrum coefficient has been computed in the 260 nm wavelength region. At a shorter wavelength, the absorption edge was achieved. The zirconium doped Nickel oxide nanoparticles exhibit strong blue emission, making them interesting for use in optical devices [19].

3.2 XRD analysis

XRD employing high intensity monochromatic radiation was used to examine the crystallinity of the NiO NPs. Scherer's formula was used to calculate the average crystalline size of Zr ion doped NiO NPs, which was 85 nm based on the FWHM (Full-width half maxima) and peak location of an XRD pattern.

$$D = \frac{0.89\lambda}{\beta \operatorname{Cos}\theta} \quad (1)$$

where D is the average crystalline size, λ wavelength in nm, e β is FWHM in radians, and, θ the diffraction angle in degrees. The largest diffraction peaks arise at 26.69, and 28.49, which correspond to the (111), and (220) planes, respectively shown in Figure 2. All of the diffraction peaks can be precisely mapped to the Face-centered cubic (FCC). JCPDS card No. 04-0835 [20] describes the crystalline structure of Zr ion doped NiO NPs. The crystalline nature of Zr ion doped NiO nanoparticles was suggested by the high intensity of the peaks.

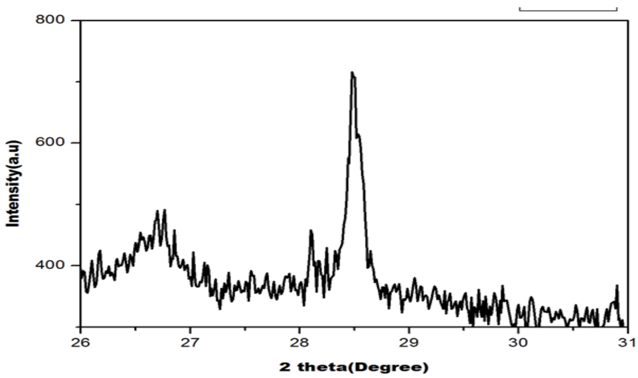

Figure 2. XRD of Zr doped NiO NPs using Acalypha indica L

3.3 FTIR ANALYSIS

Figure 3 shows the FTIR spectrum of Zr doped Nickel oxide nanoparticles produced from Acalypha indica Leaves. The existence of an O-H functional group on the surface of nanoparticles was revealed by the wide peak at 3854.43 cm^{-1}. The absorption peak at 1384.46 cm^{-1}corresponds to the aromatic amine group's C-N stretching mode. The N-H bending vibration of the main amine group is responsible for the peak at 1631.35 cm^{-1}. The C-C vibration was verified by the peak at 2362.44 cm^{-1}. Peaks at 3142.29 cm^{-1}suggested C-H stretching vibration modes in hydrocarbon chains (CH aliphatic). The stretching band vibrations for Zr bond appears at 734 cm^{-1}, important band observed at 523cm^{-1} and the band at 473.05cm^{-1} is associated with NiO [21], hereby confirmed the formation of Zr doped NiO NPs. The change in peak locations apparently suggested that the presence of metabolites such as polyphenls, flavonoids, alkaloids, and terpenoids, which are plentiful in leaf extract and create NiO nanoparticles.

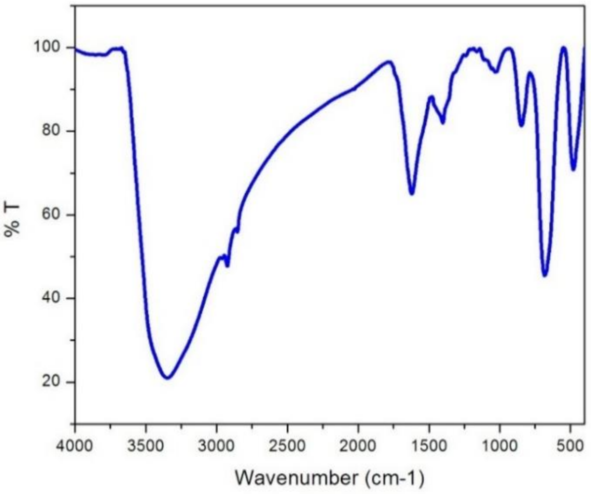

Figure 3. FTIR spectrum of Zirconium doped NiO NPs using Acalypha indica L

3.4 FESEM analysis

Field emission scanning electron microscopy (FESEM) is a strong analytical tool for performing high magnification examination on a wide range of materials and producing high resolution pictures. Field Emission Scanning Electron microscopy suite enables for the analysis of a wide range of samples, with resolution much above that of traditional light microscopy. FESEM results provides information on particle size and distribution in Figure 4, it shows the morphological dimensions of Zr doped NiO nanoparticles. From the SEM measurements, Zr doped NiO nanoparticles had slanted arrangements with each other's. Since Zr doped NiO NPs in the nanometer region as 85 nm. This continuous particles presence performing better in the electro and photo chemical applications.

3.5 Atomic force microscopy analysis

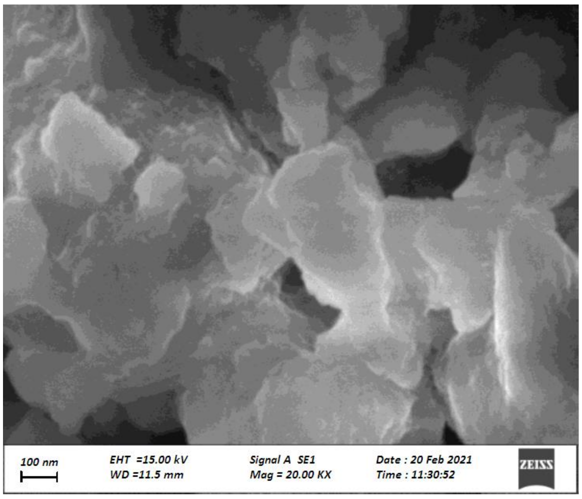

Figure 4. FESEM images of Zirconium doped Nickel oxide NPs using Acalypha Indica L

Atomic force microscopy reveals the Zr doped NiO NPs particles in the nanometers range. Nanoscan 2 easy surf instruments used to identify the nanoparticles. AFM shown the topography of the synthesized particles 2 D & 3D view in the Figure 5. It reflects with SEM images, particles are stacked with another, higher sized particles in the even sized particles, it reveals the formation of doping. From the AFM studies of Zr doped NiO nanoparticles, particles in Nano meter size and inter particles distance was low, so it has more active in applications.

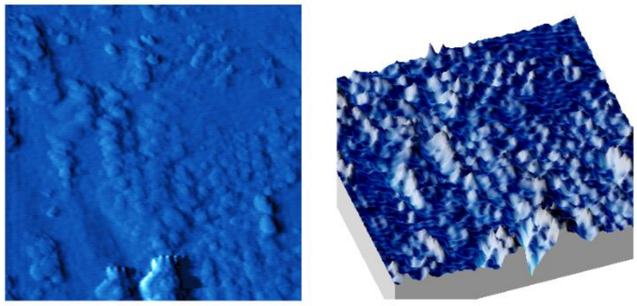

Figure 5. AFM images of Zr doped Nickel oxide NPs using Acalypha indica L.

3.6 ANTI-microbial activity

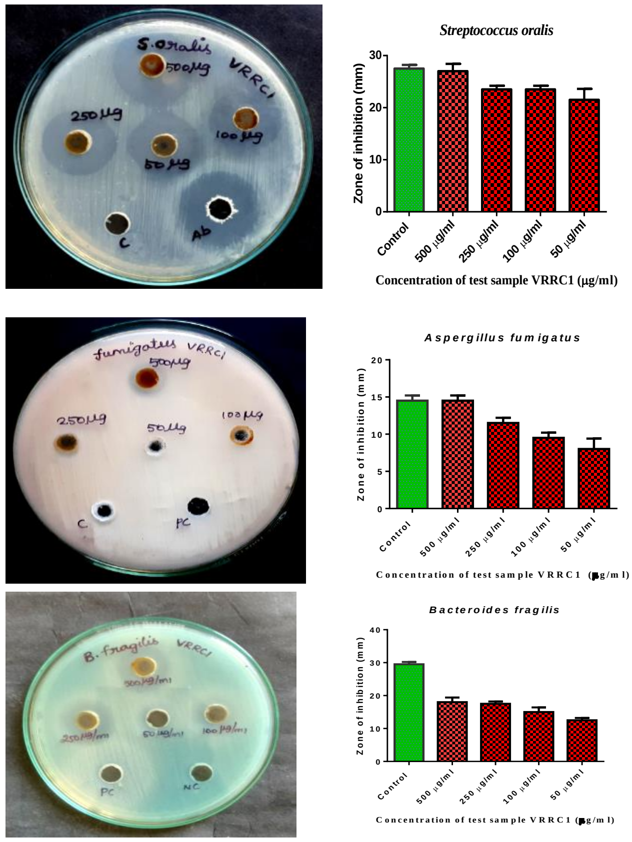

Figure 6. The antimicrobial activity of Zr ion doped NiO NPs against against the pathogens, Streptococcus oralis, Bacteriodes fragilis and Aspergillus fumigates

Agar well diffusion technique used for examined the antibacterial activity of Zr ion doped NiO NPs. Antimicrobial effect can be used for drug development, epidemiology, and treatment outcome prediction. In this application antimicrobial testing methodologies for the invitro examination of extracts and pure medicines as possible antibacterial agents. The approved method for regular bacteriological examination in many clinical microbiology laboratories is agar disk-diffusion testing, which was introduced in 1940 [22-25].

Clinically approved laboratory examined our prepared nanoparticles investigated as against the pathogens Bacteriodes fragilis, Streptococcus oralis, and Aspergillus fumigates by the following. Petri plates containing 20 ml agar media were seeded with a 24hr culture of the bacterial strain Streptococcus oralis, the fungal strain Bacteriodes fragilis, and the bacterial strain Streptococcus fumigatus. Wells were cut and various concentrations of sample Zr ion doped NiO Nps were introduced (500 g/ml, 250 g/ml, 100 g/ml, and 50 g/ml). The plates were then incubated at 37°C for 24 hours. The diameter of the inhibitory zone created around the wells was measured to determine antifungal activity. As a positive control, amphotericin B was utilised. Graph Pad Prism 6.0 software was used to compute the values (USA). Results of anti-bacterial study shown in Figure 5.

Table 1 shows that the quantity of Zirconium ion doped Nickel oxide NPs increases the zone of inhibition against the pathogens Bacteriodes fragilis, Streptococcus oralis, and Aspergillus fumigatus. At greater concentrations, zirconium doped Nickel oxide NPs are effective antibacterial and antifungal agents.

Table 1. Mean ± SD of zone of inhibition obtained by sample Zirconium doped Nickel oxide NPs against the pathogens Bacteriodes fragilis, Streptococcus oralis and Aspergillus fumigatus

S.NO.	Name of the test organism	Name of the test sample	Zone of inhibition (mm) Mean ± SD				
			500µg/ml	250µg/ml	100 µg/ml	50 µg/ml	Control (Ab)
1.	*Bacteriodes fragilis*	VRRC1	18 ± 1.0	\17.5 ±0.5	15 ± 1.0	12.5 ± 0.5	29.5 ±0.5
2.	*Streptococcus oralis*	VRRC1	27± 1.0	23.5± 0.5	23.5 ± 0.5	21.5 ± 1.5	27.5 ±0.5
3.	*Aspegillus fumigatus*	VRRC1	14.5± 0.5	11.5± 0.5	9.5 ± 0.5	8.5 ± 0.5	14.5 ±0.5

4. CONCLUSION

Acalypha indica Leaf extract was used to synthesise zirconium ion doped NiO NPs in a concise green approach. In the UV–visible spectrum, a prominent peak at 260 nm was found, confirming the formation of Zr doped NiO nanoparticles. According to Debye, Scherrer's–formula NiO nanoparticles with an average crystallographic size of 85 nm have a cubic structure as determined XRD patterns. The stacked SEM image confirms the formation of doping and is in the nano meter range. The FT-IR spectra of Zr doped NiO nanoparticles revealed a prominent band at 473.05 cm^{-1} that corresponded to the vibration of the NiO bond. Furthermore, the antibacterial activity of produced Zr doped NiO nanoparticles shown excellent inhibitory action against water-borne diseases. The produced nano crystalline NiO material in this work proved promising for prospective medicinal applications due to its competent antifungal and antibacterial capabilities.

ACKNOWLEDGEMENTS

The author is grateful to the Department of Chemistry at V.O. Chidambaram College for providing FTIR and UV and electrochemical studies. The authors are thankful to Avinashilingam University in Coimbatore for providing FESEM. The author expresses gratitude to the administration of V.O. Chidambaram College for providing the necessary supplies for this work.

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