INDIAN JOURNAL OF SCIENCE AND TECHNOLOGY

RESEARCH ARTICLE

# Structural, Optical and Antibacterial Properties of neodymium ($Nd^{3+}$) doped nickel oxide (NiO) Nanoparticles using *Sesbania grandiflora* Leaf Extract

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OPEN ACCESS
**Received:** 09.04.2020
**Accepted:** 15.12.2020
**Published:** 19.05.2021

**Citation:** Saiganesh S, Muni Sudhakar B, Thyagarajan K, Narasimha G (2021) Structural, Optical and Antibacterial Properties of neodymium ($Nd^{3+}$) doped nickel oxide (NiO) Nanoparticles using *Sesbania grandiflora* Leaf Extract. Indian Journal of Science and Technology 14(18): 1442-1451. https://doi.org/10.17485/IJST/v14i18.554

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**Funding:** None

**Competing Interests:** None

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Published By Indian Society for Education and Environment (iSee)

**ISSN**
Print: 0974-6846
Electronic: 0974-5645

## Abstract

**Objectives:** To study the eco-friendly green synthesis of neodymium doped nickel oxide nanoparticles and to study their structural, optical and antibacterial activities. **Method:** *Sesbania grandiflora* herb has been used to synthesize NiO nanoparticles. With the materials present in the plant extract, such as sugar, flavonoid, protein, enzyme, polymer, and organic acid, acting as the reducing agent the green approach takes charge in bio induction of metal ions into nanoparticles. Structural and Optical Properties of nanoparticles were studied by following XRD, SEM, EDAX, FTIR and UV-Vis-NIR (DRS). The antibacterial activity of the resultant neodymium ($Nd^{3+}$) doped nickel oxide (NiO) was tested on Gram negative and Gram positive bacteria with suitable standard. **Findings:** From the XRD analysis, it is revealed that the size of the particles is in the order of 9.2 to 23.06 nm. JCPDS data confirmed that $Nd^{3+}$ ion-doped NiO nanoparticles exhibit phases of (111), (200), (220), and (311). SEM with EDAX proved that existence of $Nd^{3+}$ ion-doped NiO nanoparticles. Cubical and spherical shapes of the nanoclusters having a size of 20-40 nm are shown by SEM analysis. Most promising peaks at 420 $cm^{-1}$ and 657 $cm^{-1}$ associated to Ni-O vibration bond and Ni-O-H stretching bond were analyzed from FTIR graph of the sample before annealing while for the sample at 500°C shows the Ni-O vibration bond at 411$cm^{-1}$. The direct band gap of $Nd^{3+}$ ion-doped NiO nanoparticles calculated as 3.08 eV at the concentration of NiONdO3 mol%. By doping with $Nd^{3+}$ ion, energy gap increases with doping concentration proved by UV-DRS spectroscopy that confirms Quantum confinement. The eco-friendly synthesized nanoparticles exhibited good antibacterial activity against pathogenic bacterial strain is an indication of antibacterial efficiency of nanoparticles. **Novelty:** Eco-friendly synthesis of NiO nanoparticles and characterization and doped nanoparticles exhibited good antibacterial activity.

**Keywords:** Green synthesis; NiO particles; Characterization; Cubical; spherical shapes; Direct band gap semiconductors; NIR laser; Antibacterial activities

# 1 Introduction

Semiconductor nanoparticles such that ZnO or NiO nanoparticles are awesome material tool for present research world because of their applications can be found in the field of catalysts, gas sensors, electrochromic film, fuel cell, magnetic materials, anode of organic light emitting diodes and thermoelectric materials(1,2). Metal oxide solar cells serve a good replacement for conventional silicon solar cells because of metal oxides are low-cost materials, wide range of optical applications. NiO is a P-type semiconductor with a wide band gap between 3.5 eV and 4.0 eV(3,4). Electronic devices are being increasingly used in our lives. Fossil fuels are being replaced by renewable energies, a field that increasingly uses rare earth elements. These elements are used in electric cars and wind turbines. Although procurement challenges and high prices lead producers to seek alternatives, rare earth elements are still being used in numerous technologies and because of their demand, the health effects of these powerful magnets must be addressed along with their environmental impacts. In recent years, biosynthesis of nanoparticles has received considerable attention due to the growing need to develop clean and nontoxic chemicals, low-cost approaches, eco - friendly solvents and renewable materials and so preferable method for NPs synthesis can be done by green synthesis method(5,6). In the reported journal, NiO nanoparticles exhibits super paramagnetic behavior, when magnetization studies are approved. It is then evaluated for cytotoxic activity towards A549 cell culture, antibacterial activity and photo catalytic degradation (PCD) of 4 chlorophenol (4 CP), which is known as the endocrine disrupting chemical (EDC).

The purpose of this work is the synthesis of NiO nanoparticles by the chemical precipitation process in the presence of sodium hydroxide, which is a simple way and low in cost since the starting materials are few and inexpensive.

# 2 Method of synthesis

## 2.1 Synthesis of NiO nanoparticles

The leaf of *Sesbania grandiflora*, a plant that belongs to family Fabaceae cultivated in all over India, is used. The plant has synonym *Agati grandiflora* and commonly called as Hummingbird tree or Butterfly tree. It contains alkaloids, phenols, flavonoids, carbohydrates, saponins, phytosterols, Triterpenes, Amino acids and Alcohols. Leaves are used to disinfect throat and cure kidney diseases. Its Telugu name is Avisaaku frequently available in Andhra Pradesh state in the region of Rayala Seema districts in India.

The plant leaf was collected at village nearer to our hometown Srikalahasti. Initially, the fresh leaves of 10 gm *Sesbania grandiflora* was obtained and are shade dried over for two days and boiled in a Pressure cooker with water of 500 ml over sufficient boiling. After deep mixing of plant with water, cooled and residual extract is filtered with Whatman filter paper to transfer into 500 ml glass beaker.

Nickel chloride hexa hydrate ($NiCl_2.6H_2O$) (Sigma Aldrich 99.99%), Neodymium (Nd) (used as doping agent), and NaOH were used as the preparation materials for the synthesis of NiO nano particles (NPs). At first, 2g of $NiCl_2.6H_2O$ and 60 mg of Nd was dissolved in 250 ml of distilled water to get a certain molar concentration at room temperature by constant high-speed stirring of 500 rpm. The doping agent was varied as 02%, 03%%, 04%%, 05% and 06% by weight percentage of NiONd samples and added Distilled water to samples as a dispersing solvent. After dissolved binary compound aqua solution (takes approximately 10 min), plant extract was added to the solution in the ratio of 1:10 (25 ml). Afterwards, the 2M of NaOH was added by drop wise to both the mixture (binary compound aqua solution and plant extract) of solution until the pH of becomes 12. Then, it is placed in a magnetic stirrer at a minimum speed for 6 hrs. Finally, green gel is formed and the obtained greenish gel was heated at $80^0$C for 24 hours to obtain precipitate.

The precipitate was washed with water and Ethanol repeatedly by centrifugation to remove the residues or create it free from impurities. After that the dried greenish type powder samples were annealed at $500^0$C temperature for 2 h to obtain NiO nanoparticles. Due to annealing, the color of greenish type powder samples changed to black powder.

Prior to the NiO NPs preparation, extract was prepared from the extrapolation of plant leaves. The extract was obtained from boiling the 10g of plant leaves in 500 ml of water. Finally, 350 ml of leaves extract was obtained, and the extract stored in a bottle for future use.

## 2.2 Characterization techniques

X-ray diffraction (XRD) analysis was used to characterize the crystalline nature of the NiO nanostructures, and the Bragg's diffraction peaks were obtained using PANalyticalX'Pert PRO diffractometer equipped with Cu-K$\alpha$ radiation of 1.54 $A^O$. The NiO nanoparticles XRD spectrum was observed in the range of 10 — 80°. This spectrum shows the sharp intensity of the crystalline nature of the samples. The morphology of the prepared samples of nanostructures were characterized by scanning electron microscopy (SEM, S4800 Hitachi model). Using the SEM with EDAX the samples were analysed in the range 10-50 nm at room temperature.

The absorption spectra of $Nd^{3+}$ ion doped with different concentration of NiO nanoparticles were analyzed with a UV-Vis-NIR absorption spectrophotometer (Thermo Scientific Genesys 10S, USA) with a resolution of 1nm, in the range of 200-2500 nm at room temperature.

### 2.3 Antibacterial properties

Antibacterial action of NiO nanoparticles was studied by zone of inhibition method. For this method. The bacterial strains such as *E. coli* and *Bacillus* spp, obtained from cultural collection centre, were used. The pure strains of these two species of bacteria were inoculated in the Luria broth (Liquid phase) in 100 ml conical flasks separately and cultured for 24 hr. These flasks were incubated in an incubator for overnight at $37^0$C(7,8). The overnight active *E. coli* and *Bacillus* spp. cultures were seeded into the Luria agar medium by using spread plate techniques. Later in the Luria agar plates, cavities were made with the help of the metallic sterile borer. All the cavities were filled with 100$\mu$l of $Nd^{3+}$ ion doped with NiO nanoparticles solution and the Petri plates were then incubated at $37^0$C in the incubator for overnight incubation.

## 3 Results and analysis

### 3.1 Structural properties

*i) XRD analysis*

The XRD spectral analysis of $Nd^{3+}$ ion doped with NiO nanoparticles was identified the sharp peaks in the crystalline nature, as shown in Figure 1 form this XRD analysis studied the structure and size of the NiO nanoparticles. The size of the particle at different doping concentration can be calculated from the x-ray peaks of $Nd^{3+}$ ion Doped NiO nanoparticles by the Scherer formula(9).

$$D = \frac{K\lambda}{\beta_{hklcos\theta}} \tag{1}$$

Where k = Scherer constant (0.89), the wavelength of Cu$k_\alpha$ line in the instrument used is $\lambda$=1.5406$A^o$, full width at half maximum ($\beta_{hkl}$) can be calculated from the graph between intensity and 2$\theta$, diffraction angle of x-rays on Bragg's plane is $\theta$ will be known in the XRD instrument.

Strain due to crystal imperfection in nanoclusters can be estimated from William-Hall formula(10) is given by

$$\varepsilon = \frac{\beta_{hkl}}{4tan\theta} \tag{2}$$

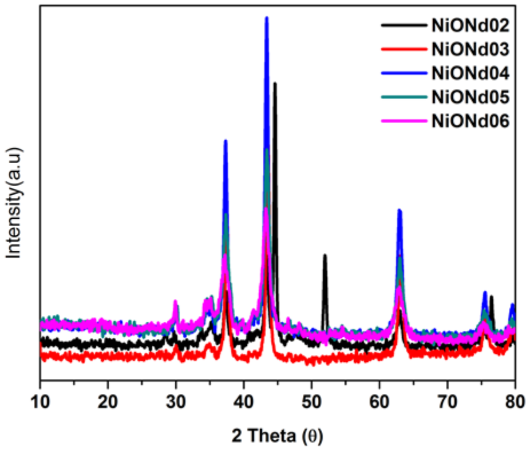


**Fig 1.** X-ray peaks of Nd3$^+$ ion doped with different concentrations of NiO nano particles.

**Table 1.** X-Ray peaks analysis for $Nd^{3+}$ ion doped NiO nanoparticles.

| S.No | Sample | $2\theta$ values at phase |  |  |  | Average Particle Size (D, nm) | Average Strain ($\varepsilon$) |
|---|---|---|---|---|---|---|---|
|  |  | (111) | (200) | (220) | (311) |  |  |
| 1 | Nd 02 | 37.35 | 44.6 | 62.92 | 76.48 | 23.06 | 0.0835 |
| 2 | Nd 03 | 37.5 | 43.55 | 63.13 | 75.59 | 17.59 | 0.0996 |
| 3 | Nd 04 | 37.33 | 43.38 | 62.98 | 75.5 | 18.14 | 0.0880 |
| 4 | Nd 05 | 37.38 | 43.41 | 62.98 | 75.39 | 12.83 | 0.1224 |
| 5 | Nd 06 | 37.19 | 43.27 | 62.89 | 75.24 | 9.2 | 0.1750 |

From the Table 1, the average particle size almost lowers with enhancing doping concentration, while average strain moves up. The data values match with standard JCPDS data card number 04-0835. The XRD spectrum of the $Nd^{3+}$ ion doped NiO nanoparticles exhibit the different crystalline phases, such as (111), (200), (220) and (311), in which the particle size is in range of 20-50nm.The hkl parameters show the lattice vibrations of the NiO nanoparticles into the structural arrangements of the sample. It is a good analysis of the NiO materials.

### *ii) FTIR analysis of NiONd nanoparticles*

The FTIR spectroscopy is an investigation method that provides the structural studies to reconnoiter the important and functional groups in crystalline and non-crystalline matrices.

The vibrational modes of functional groups in $Nd^{3+}$ ion-doped NiO nanoparticles were confirmed by an Attenuated Total Reflectance (ATR) mode FTIR spectrophotometer in the range of 400 – 4000 $cm^{-1}$. The observed broad bands are due to the higher degeneracy of vibrations states, thermal broadening of the lattice dispersion, and mechanical strength of the production of powder samples, and corresponding band assignments. Figure 3 shows the several peaks specifying the local structure of the $Nd^{3+}$ ion-doped NiO nanoparticles. The FTIR spectrum shows the characteristics peaks at 418.57 $cm^{-1}$, 677.04 $cm^{-1}$, 1629.90 $cm^{-1}$, 2991 $cm^{-1}$, 3309 $cm^{-1}$. The band at 418.57 $cm^{-1}$ reveals the presence of NiO. The peak around 3309 $cm^{-1}$ shows the presence of $OH^-$ bonds. The absorption bands at 1042$cm^{-1}$ and 1406 $cm^{-1}$ reveals that the existence of carbonates and 2991 $cm^{-1}$ exhibits C-H stretching mode. At room temperature, the most promising peaks at 418.57 $cm^{-1}$ and 677.04 $cm^{-1}$ associated with Ni-O vibration bond and Ni-O-H stretching bond as shown in .

After annealing the sample at 500°C, the absorption band at 1055 $cm^{-1}$ and 1419 $cm^{-1}$ primarily proves that the carbonates group is associated with the sample as shown in Figure 5. The significant peak at 411$cm^{-1}$ throws light on the existence of Ni-O bond in the sample(11–13). It implies that the presence of hydroxyl in the precursor and the broad absorption around 767 $cm^{-1}$ is assigned to the band stretching vibrations.

The serrated absorption bands in the region of 1000-1500 $cm^{-1}$ are assigned to the O-C = O symmetric and asymmetric stretching vibrations and the C-O stretching vibration, but the intensity of the band has weakened, which indicated that the ultrafine powers tend to strong physical absorption to $H_2O$ and $CO_2$.

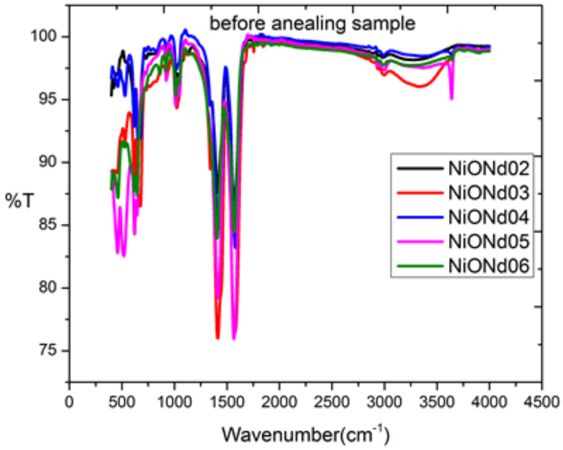


**Fig 2.** Before annealing the Nd3+ ion doped NiO nanoparticles of FTIR spectrum.

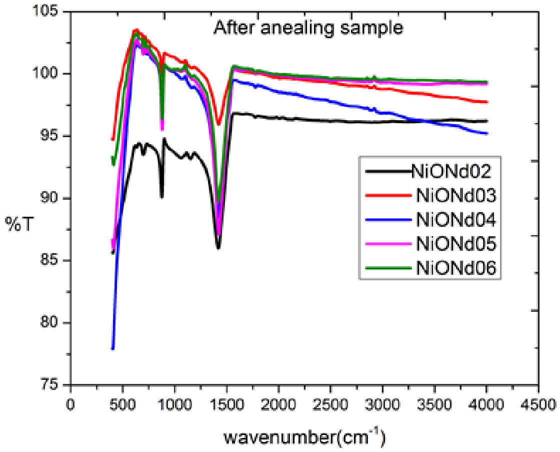


**Fig 3.** After annealing the $Nd^{3+}$ ion doped with different concentration of NiO nanoparticles of FTIR spectrum.

## 3.2 Morphological properties

### SEM with EDAX analysis of NiONd samples

The Shape of the nanoparticles appears to be spherical and Cubical by analyzing SEM with EDAX analysis as shown in Figure 4 (a) and (b). In the past, researchers evaluated that the spherical nanoparticles can be obtained by green synthesis method(14). It was proved from present observation of SEM image, the size of the nanocluster was estimated as 20-40 nm from the SEM analysis, which is shown in Figure 4(b). From EDAX analysis confirmed that $Nd^{3+}$ ion doped into NiO nanoparticles and is shown in Figure 4(a). EDAX analysis for $Nd^{3+}$ ion doped NiO nanoparticles is presented in Table 2. The nanoparticles size calculated in the SEM images was approximately in good agreement with the particle size calculated from the XRD spectrum.

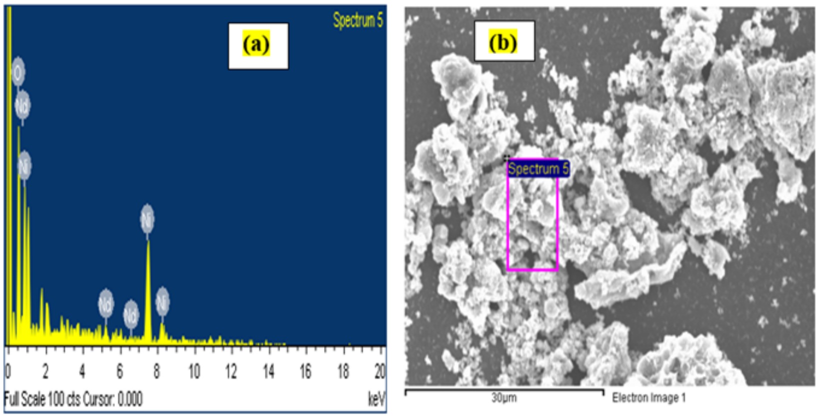


**Fig 4.** SEM with EDAX images of $Nd^{3+}$ ion doped NiO nanoparticles. (a) EDAX image shows the NiO nanoparticles. (b) SEM image of NiO nano particles in the range of 20 - 40 nm.

**Table 2.** EDAX analysis for $Nd^{3+}$ ion doped NiO nanoparticles.

| Element | Weight% | Atomic% |
|---|---|---|
| O | 25.89 | 57.10 |
| Ni | 69.48 | 41.76 |
| Nd | 4.63 | 1.13 |

### 3.3 Optical properties

The quantity of optical absorption and predominantly the absorption spectra is an important investigation of the optical induced transitions and for analyze the information about the band structure and optical band gap of non-crystalline materials. The UV-Visible spectrum of $Nd^{3+}$ ion doped NiO nanoparticles as shown in Figure 5. In UV-Visible region absorption spectrum, characteristic peaks of $Nd^{3+}$ ion doped NiO nanoparticles observed at 380 nm, 380 nm, 383 nm, 383 nm and 389 nm for the samples of NiONd02%, NiONd03%, NiONd04%, NiONd05% and NiONd06%. Due to quantum confinement absorbed wavelength shifts to lower side with raising doping concentration.

The NIR spectrum of NiONd03% doped NiO nanoparticles as shown in Figure 6, it is clear that absorption band of trivalent rare earth metal ions ($Nd^{3+}$) originated at NIR region due to 4f-4f transition. This material exhibits absorption band at 808 nm and 872 nm due to the Transitions ${}^4I_{9/2}\rightarrow{}^4IF_{3/2}+{}^2H_{9/2}$ and ${}^4I_{9/2}\rightarrow{}^4F_{3/2}$ respectively. The assignment of absorption transitions has been located according to the earlier studied $Nd^{3+}$ ion doped NiO nanoparticles.

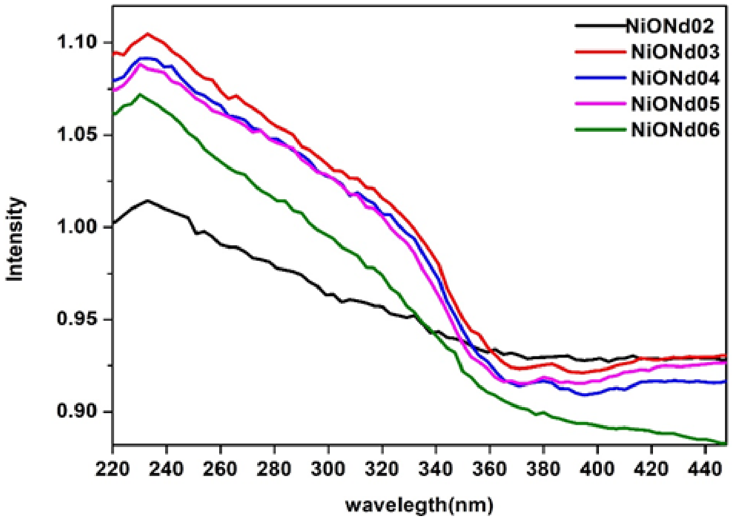


**Fig 5.** UV-Visible spectrum of $Nd^{3+}$ ion doped NiO nanoparticles.

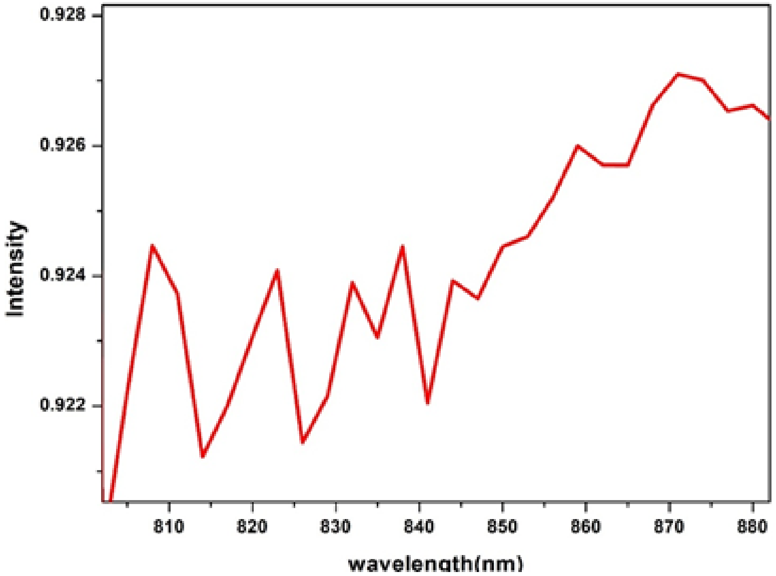


**Fig 6.** NIR spectrum of NiONd03 doped NiO nanoparticles**.**

It is observed that the maximum strongest absorption edge of the $Nd^{3+}$ ion doped NiO nanoparticles appears to be at 233nm for NiONd03% doped sample. This absorption in the UV region is attributed to the band gap absorption in NiO. The energy gap of $Nd^{3+}$ ion doped NiO nanoparticles can be determined by using Tauc relation (15) and is presented in Table 3 .

$$(\alpha h\nu)^n = A\,(h\nu - E_g) \tag{3}$$

Where, n is the number dependence upon the nature of the transition process and A is Coefficient of absorbance. For direct transition n=2, for indirect n=1/2. So, the direct band gap of $Nd^{3+}$ ion doped NiO nanoparticles can be obtained by extrapolating the linear portion of the $(\alpha h\upsilon)^2$vs $h\upsilon$ curve to the energy axis shown in Figure 7. The band gap of $Nd^{3+}$ ion doped NiO nanoparticles calculated from this plot is in between 2.05 to 3.08ev and increases with raising doping concentration of $Nd^{3+}$ ion (16).

**Table 3.** $(\alpha h\upsilon)^2$ versus $h\upsilon$ graphs analysis data for $Nd^{3+}$ ion doped NiO nanoparticles

| Sl. No | Sample | Energy gap (eV) |
|---|---|---|
| 1 | NiONd02% | 2.53 |
| 2 | NiONd03% | 3.08 |
| 3 | NiONd04% | 2.86 |
| 4 | NiONd05% | 2.05 |
| 5 | NiONd06% | 2.15 |

A red shift was observed in band gaps $Nd^{3+}$ ion doped NiO. Amount of this red shift is higher for low concentration of dopant. The highest shift was founded for NiONd05% sample shown in Figure 8, which is in good contract with our results. It is well acknowledged that semiconductors through nanoscale size illustration a red shift in their spectra outstanding to the quantum confinement effects. This suggests that with the increases the band gap value, the particle size became smaller (17). The increasing trends of the band gap energy on decrease in particle size well presented for the synthesized samples as shown in Figure 7. This effect likely due to the chemical defects or vacancies present in the intergranular regions, generating a new energy level to reduce the band gap energy. No linear relation was found for n =1/2 suggesting that the synthesized NiO nanoparticles are semiconducting with direct transition at this energy (18).

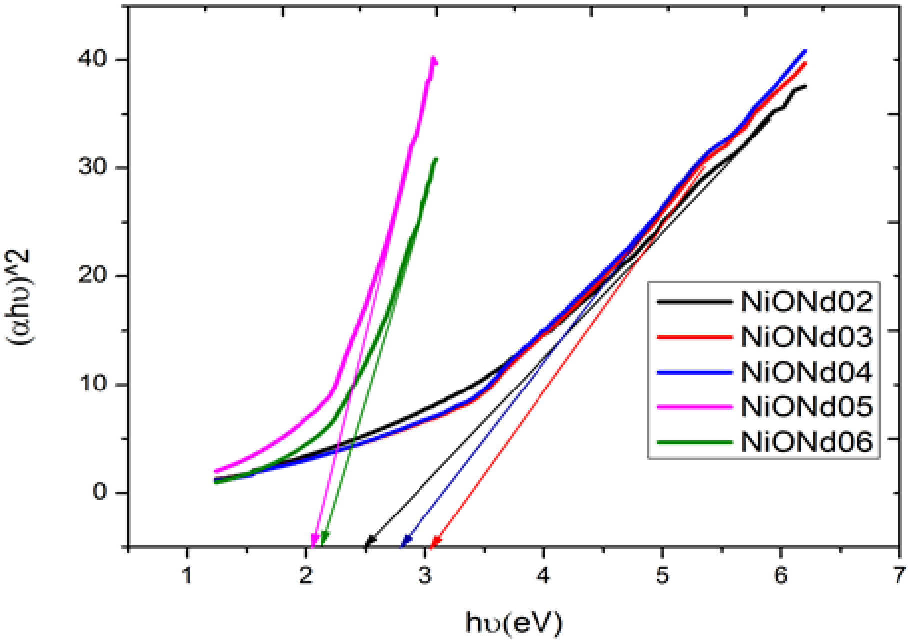


**Fig 7.** Energy gap of $Nd^{3+}$ ion doped with different concentration of NiO nanoparticles

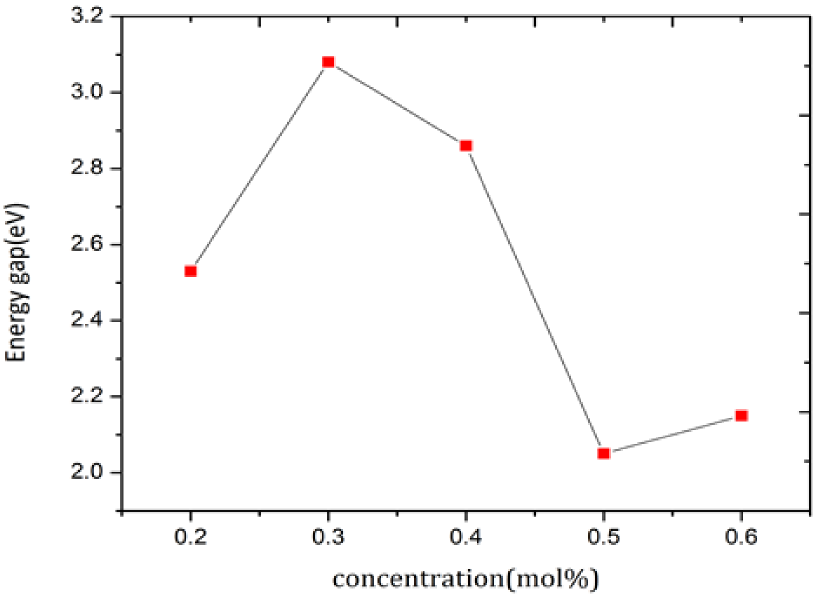


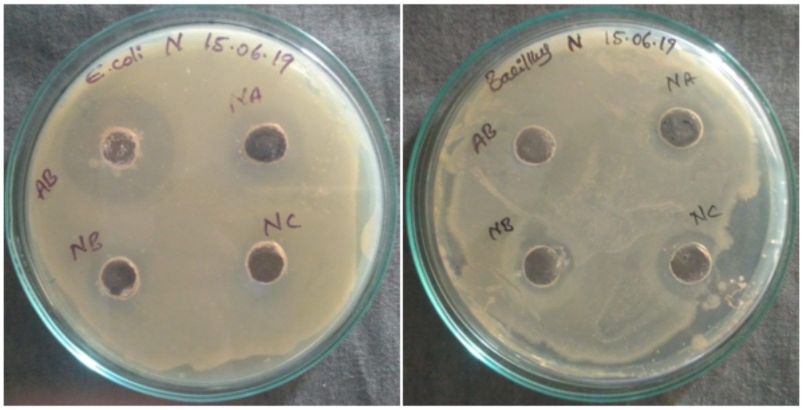
**Fig 8.** Energy gap Vs Concentration graph.

### 3.4 Antibacterial properties

Antibacterial action of NiO nanoparticles was obtained by zone of inhibition method shown in Figure 9. Antimicrobial activity and antifungal activity of nanoparticles depends on physiochemical properties of nanoparticles and type of microbial pathogen, they can seize the functions of cell of *E. coli* significantly. *Bacillus* spp. contain thicker peptidoglycan cell wall layer so the toxicity of NiO nanoparticles is lesser to them compare to *E. coli* which was shown in Table 4 and Figure 10.

By observing the data about inhibition zone, Nd doped NiO nanoparticles exhibited good antibacterial activity against gram positive and gram-negative bacterial strains, *Bacillus* spp. and *E. coli* grown in nutrient agar medium (Table 4).

Anti-bacterial activity enhances by raising doping concentration as shown in the Figure 9. The standard antibiotic streptomycin was used as positive control. The maximum size of the inhibition zone observed for the sample NiONd 04% against gram positive and gram negative bacterial strains, *Bacillus* spp. and *E. coli* grown in nutrient agar medium of the value 1.05 cm and 1.20 cm; it was more similar function as for as standard antibiotic streptomycin shown in Figure 10



9(a) 9(b)

**Fig 9.** Antimicrobial activity of $Nd^{3+}$ ion doped with NiO nanoparticles against gram positive and gram-negative bacterial strains. Such as (a) *E. coli* and (b) *Bacillus* spp.

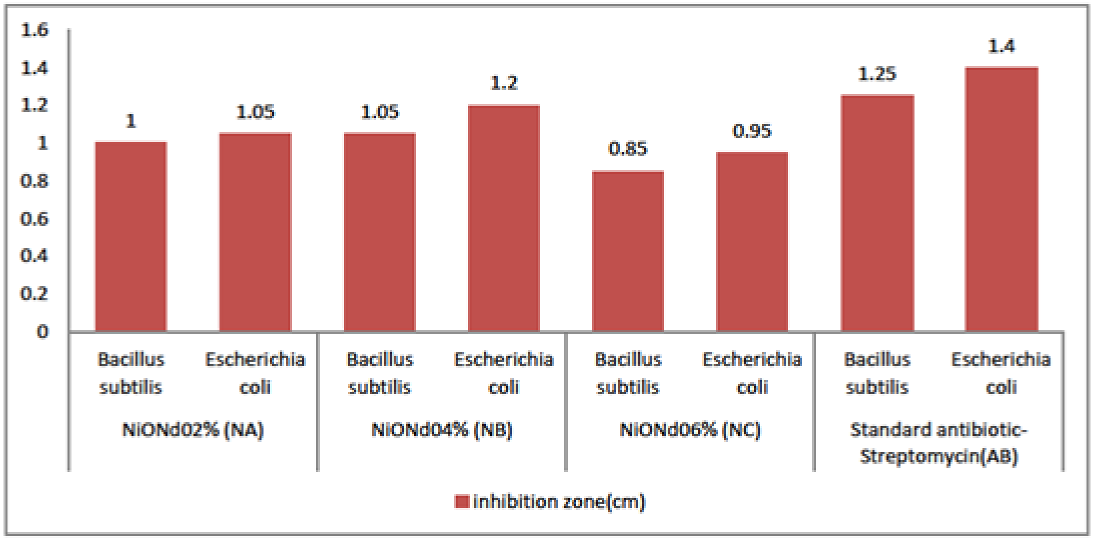


**Fig 10.** Zone of Inhibition

**Table 4.** Antibacterial activity of $Nd^{3+}$ ion doped NiO nanoparticles.

| Sample | Microorganisms | Zone of inhibition (cm) |
|---|---|---|
| NiONd02% (NA) | *Bacillus subtilis* | 1.0 |
| | *Escherichia coli* | 1.05 |
| NiONd04% (NB) | *Bacillus subtilis* | 1.05 |
| | *Escherichia coli* | 1.2 |
| NiONd06% (NC) | *Bacillus subtilis* | 0.85 |
| | *Escherichia coli* | 0.95 |
| Standard antibiotic Streptomycin (AB) | *Bacillus subtilis* | 1.25 |
| | *Escherichia coli* | 1.4 |

## 4 Conclusions

Neodymium doped nickel oxide nanoparticles were synthesized through the eco-friendly procedure by *Sesbania grandiflora* extract and characterized by standard instrumentation (XRD, SEM, EDAX, FTIR and UV-Vis-NIR (DRS)); the synthesized nanoparticles were showed antibacterial activity against gram-negative bacteria, *Escherichia coli* (1.2cm) than the gram-positive bacteria, *Bacillus* spp. (0.85cm) at NiONd04% sample. For a rare earth metal ion like neodymium, the antibacterial activity has not been researched much. So the neodymium doped nickel oxide nanoparticles can be used in current nanomedicine and also can be used in alternative to the bleaching process of waste water treatment.

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