



## A NOVEL ONE STEP METHOD TO SYNTHESIS AND CHARACTERIZATION OF ECO-FRIENDLY NICKEL OXIDE (NiO) NANOPARTICLES AND ANTIBACTERIAL ACTIVITY

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### ABSTRACT

Green synthesis techniques make use of moderately pollutant free chemicals to synthesis nanomaterials and embrace the use of benign solvents such as water, natural extracts. Green chemistry seeks to reduce pollution at source. Though physical and chemical methods are trendier for nanoparticles synthesis, the biogenic fabrication is a better choice due to eco-friendliness. Nanoparticles due to their smaller size and large surface to volume ratio exhibit remarkable novel properties and methodical applications in the field of biotechnology, sensors, medical, catalysis, optical devices, DNA labeling, drug delivery and they are rewardingly treated as a bridge between bulk material and atomic and molecular structures. To Synthesize the Ni (OH)<sub>2</sub> precursors from various acids like Cinnamic Acid and Oxalic acid and Asteracea Sirreu Sunrise Flower Extract. To characterize the various Ni (OH)<sub>2</sub> precursors with the help of analytical instruments such as UV-Vis, IR. To Bio synthesize the NiO nanoparticles from various precursors. To characterize the NiO nanoparticles with the help of analytical instruments such as UV-VISIBLE, FT-IR, XRD, SEM with EDAX analysis. To analyze the antibacterial activity of the NiO nanoparticles with both gram positive and gram-negative bacteria such as Gram-positive *Staphylococcus aureus* *Streptococcus Pyogenes* Gram negative *Proteus Vulgaris*, *Klebsiella Pnemoniae* & *Escherichia coli*.

### KEY WORDS

NiO, UV-Visible, XRD, FT-IR and Antimicrobial activity.

### 1. INTRODUCTION

In the last few decades, many researchers were interested in preparing and studying the nanostructured materials that have unique physical and chemical properties. These properties are dependent on size, shape and crystallography of nanomaterials. Therefore, the synthesis of nanostructured materials with controlled size and shape becomes very important [1]. Currently, unlike the chemical and physical synthetic

methods, biological systems provide a novel idea for the production of nano materials. Several plant extracts and even microorganisms from bacteria to fungi were utilized to synthesize inorganic materials in nano scale either intra or extra cellularly [2]. NiO is considered one of the most commonly used metal oxides for a wide range of applications where NiONPs were shown to have novel optical, electronic, magnetic, thermal, and mechanical properties. In addition, it was used in many

potential applications such as in battery electrodes, gas sensors, photo electronic devices.... etc [3-5].

Based on the above-mentioned literature, it can be concluded that as synthesized NiO nano powders produced by the Green synthesis method have an amorphous structure and are relatively small in size up to 10 nm. At the same time, they can contain particles with various concentrations of the components different from the initial concentration. It should be noted, that amorphous NiO nanoparticles were also obtained by dissolving Ni (OH)<sub>2</sub> precursor salts Ni (OH)<sub>2</sub> precursors from various acids like Cinnamic Acid and Oxalic acid and *Asteracea Sirreu* Sunrise Flower Extract [6,7]. The SEM analysis of as synthesized NiO nanopowder showed agglomerated particles 0.5–20 µm in size. In all the studies discussed above, except Ref. [8-13]. This fact, together with the manifested differences of structural data in the literature, motivated us to study the structure and morphology of as synthesized NiO Green synthesis method using *Asteracea Sirreu* Sunrise Flower Extract. The as synthesized nanoparticles were characterized using powder XRD and SEM analysis [14-19].

## 2. EXPERIMENTAL TECHNIQUES

### 2.1 Materials

Nickel Sulphate 6 H<sub>2</sub>O, Nickel Chloride 6 H<sub>2</sub>O, Cinnamic Acid, Oxalic Acid was used as the introductory material was supplied by Sigma Aldrich chemicals. A fresh leaf of *Asteracea Sirreu* Sunrise flower was washed thoroughly with double distilled water, grinded and was filtered through Whatman filter paper was used for further studies. Then the Flower were nicely crushed in mortar pestle, after that the nicely crushed Flower were transferred into a centrifuge tube and centrifuged (Optima L-100XP Ultra centrifuge at a speed of 10000 - 11000 rpm for 10 min at 4 to 5°C). After centrifugation,

the supernatant was filtered using Whatmann paper and filtrate was used for the synthesis of NiO.

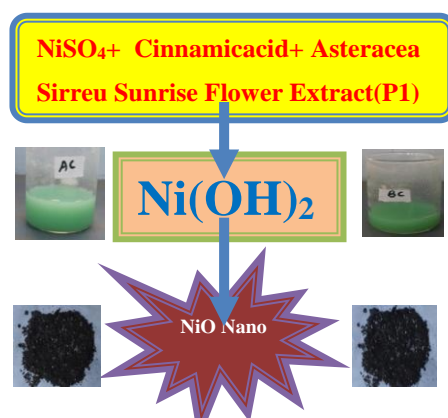
### 2.2 Synthesis of NiO Nanoparticles

#### 2.2.1 Synthesis of NiO Nanoparticles Using *Asteracea Sirreu* Sunrise Flower Extract from Cinnamic Acid P1 Precursor.

NiO nanoparticle was prepared by Green synthesis method. In synthesis to 10 mL of 0.1M Nickel sulphate solution was taken and made upto 100 mL and to the same 0.2 M of Cinnamic Acid was added to aqueous solution. To these solutions 10mL of *Asteracea Sirreu* Sunrise Flower Extract was added and stirred for about 6 hrs at room temperature. The Light Green color residue thus obtained was collected in a previously cleaned, washed and dried silica crucible. It was heated to 400°C for 2 h in a muffle furnace. The black colored Nickel oxide (NiO) nanoparticles thus obtained was collected, preserved and used for further characterization and applications. Similar method as adopted for the synthesis of NiO nano-particles using Nickel Chloride as precursor [20].

#### 2.2.2. Synthesis of NiO Nanoparticles Using *Asteracea Sirreu* Sunrise Flower Extract from Oxalic Acid P2 Precursor.

NiO nanoparticle was prepared by Green synthesis method. In synthesis to 10mL of 0.1M Nickel Sulphate solution was taken and made up to 100 mL and to the same 0.2 M of Oxalic Acid was added to aqueous solution. To these solutions 10mL of *Asteracea Sirreu* Sunrise flower extract was added and stirred for about 6 hrs at room temperature. The Green color residue thus obtained was collected in a previously cleaned, washed and dried silica crucible. It was heated to 400°C for 2 h in a muffle furnace. The black colored Nickel oxide (NiO) nanoparticles thus obtained was collected, preserved and used for further characterization and applications.



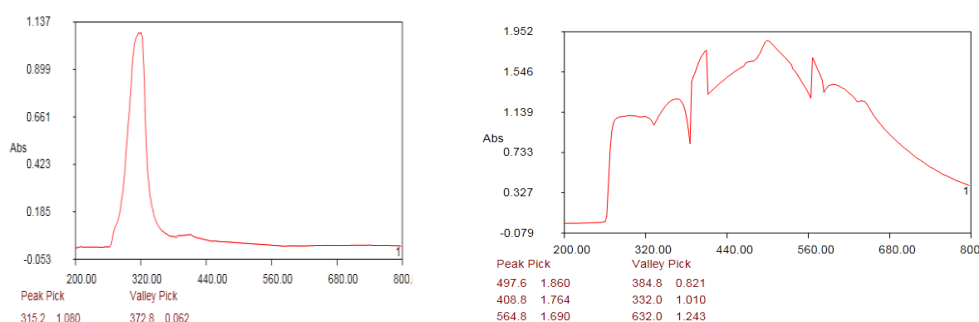
**Scheme 1. The Synthesis of Nickel Oxide (NiO) nanoparticles by Green Method.**

### 3. RESULTS AND DISCUSSION

#### 3.1 UV-VIS spectroscopy

Probably one of the most universally utilized spectroscopy techniques, the adsorption of electromagnetic radiation in the range from ultraviolet to visible is still a versatile research tool. Just in the case of FT-IR, UV-VIS can be used to analyze certain compounds used in the functionalization of nanoparticles for dispersions and applications. Changes in the UV part of the spectrum can commonly be contributed to charge transfer bands between a surface metal cation and the functional ligand. From the spectra obtained by UV-VIS Spectrophotometer it is known that

a smooth and narrow absorption bands were obtained [21]. This spectrum is recorded immediately after the synthesis of particles. The figure 8 shows the UV absorption peaks at 408 nm respectively, which proves the formation of the nickel nanoparticles in the solution. The initial green color of the solution turned into a black precipitate, the shifting in color is due to the surface Plasmon resonance. Metals possess Surface Plasmon resonance band in visible region due to free electrons, which give such intense colors. These properties observed in nickel are due to the presence of free electrons [22].



**Figure - 1 UV-Visible Spectra of Ni (OH)<sub>2</sub> compound P1 and NiO nanoparticles N1**

#### 3.2 Band Gap Energy Value of NiO nanoparticles in the Powder Form using a UV/Vis Spectrometer.

The measurement of the band gap of materials is important in the semiconductor, nanomaterial and solar industries. This note demonstrates how the band gap of a material can be determined from its UV absorption spectrum. The values are shown in the given table 4-5. With similar experimental conditions and accessories, band gap energy values for various Precursor and Nano-powder nanomaterials can be calculated. With this, the

quality of NiO also can be determined compared precursor. Various other semiconductor nanomaterials can also be subjected to the experiment method matched theoretical value [23].

$$\text{Band Gap Energy (E)} = h * C / \lambda$$

$$h = \text{Planks Constant} = 6.626 \times 10^{-34}$$

$$C = \text{Speed of light} = 3.0 \times 10^8$$

$$\lambda = \text{Cut off wavelength}$$

$$\text{Where } 1 \text{ eV} = 1.6 \times 10^{-19} \text{ (J)}$$

**Table – 1 Shows Band Gap Energy for various Precursors**

Precursor (P)	$\lambda$	E	eV
P1	315	6.3064	3.9415
P2	315	6.3064	3.9415

**Table – 2 Shows Band Gap Energy for NiO Nanoparticles**

NiO Nanoparticles	$\lambda$	E	eV
N1	408	4.8625	3.0390
N2	384	5.1658	3.2286

### 3.3 POROSITY

Porosity is one of the most important rock properties in describing porous media. It is defined as the ratio of pore volume to bulk volume of a rock sample. The porosity of a rock is the fraction of the volume of space between the solid particles of the rock to the total rock volume. The space includes all pores, cracks, vugs, inter- and intra-crystalline spaces. The porosity is conventionally given the symbol  $f$ , and is expressed

either as a fraction varying between 0 and 1, or a percentage varying between 0% and 100%.

$\phi = \text{pore volume} / \text{bulk volume}$

When core is saturated with water, volume of absorbed water is equal to pore volume of core sample.

**weight of absorbed water = wet weight - dry weight**

**Volume of absorbed water = pore volume = weight of water \* density**

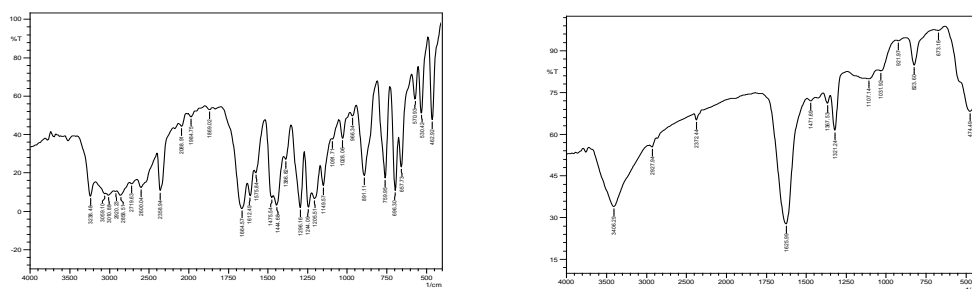
**Table – 3 shows the Porosity of the Precursor and NiO nanoparticles.**

COMPOUND	POROSITY %
P1	0.170
P2	0.210
N1	0.150
N2	0.205

### 3.4 IR – SPECTRA ANALYSIS

Figure 2 shows FT IR spectroscopy is a useful tool to understand the functional group of any organic molecule. Various bands were observed in the FTIR spectra. The position and number of absorption bands not only depend on crystal structure and chemical composition but also on crystal morphology. The broad band observed around  $3500 \text{ cm}^{-1}$  and  $1600 \text{ cm}^{-1}$  corresponds to O-H stretching vibration due to the absorbed water on the surface of the samples. The series of absorption in the current FTIR Spectra correspond to the impurities present in the samples. The peak at  $667 \text{ cm}^{-1}$  is attributed to O=C=O bending vibration. The band in the region  $670\text{-}1000 \text{ cm}^{-1}$  is due

to C-H bending of alkene while at  $1660 \text{ cm}^{-1}$  is due C=C stretch of the same. The prominent peaks at  $1071 \text{ cm}^{-1}$  and  $1156 \text{ cm}^{-1}$  are because of C-O stretch (alcohol). The absorption bands observed around  $1280 \text{ cm}^{-1}$  and  $1330 \text{ cm}^{-1}$  correspond to C-O stretching (acid). The peaks around  $1383 \text{ cm}^{-1}$  and  $1460 \text{ cm}^{-1}$  reveal the presence of -C-H bending vibration of alkane and that around  $2850\text{-}3000 \text{ cm}^{-1}$  reveal its C-H stretching vibrations. The broad absorption band in the region of  $3400 \text{ cm}^{-1}$  corresponds to the O-H stretching vibrations of water present in the powder sample. The intense absorption peak at  $\sim 500$  to  $600 \text{ cm}^{-1}$  is related to the stretching vibrations of Ni-O bond [24-27]. The results further confirm XRD results of the spectra.



**Figure -2 FT IR Spectral graph for Ni (OH)<sub>2</sub> P1 and NiO Nanoparticles N1**

### 3.5 XRD – ANALYSIS

Powder X-ray Diffraction (XRD) is one of the primary techniques used by mineralogists and solid-state chemists to examine the physicochemical makeup of unknown materials. X-ray diffraction is one of the most important characterization tools used in solid state chemistry and materials science. XRD is an easy tool to determine the size and the shape of the unit cell for any compound. Powder Diffraction Methods is useful for Qualitative analysis (Phase Identification), Quantitative analysis (Lattice parameter determination & Phase fraction analysis) etc. Diffraction pattern gives information on translational symmetry - size and shape of the unit cell from Peak Positions and information on electron density inside the unit cell, namely where the atoms are located from Peak Intensities. It also gives information on deviations from a perfect particle, if size is less than roughly 100 nm extended defects and micro strain from Peak Shapes & Widths.

Figure shows the XRD patterns of samples synthesized by coprecipitation methods with dry temperature  $T_d$  of 400°C for 2 h. The spectra are almost equal to the typical XRD spectra of NiO nanoparticles reported from other experiments. In all our XRD patterns nine peaks are observed around  $2\theta = 32.14, 33.46, 37.6, 47.66, 58.54, 63.52, 69.95, 72.23, 74.24$  which correspond to (100), (002), (101), (102), (110), (103), (200), (112) and (201), respectively. For each sample, all observed peaks can be indexed as the hexagonal wurzite structure of NiO with having space group  $P6_3mc$ .

The values of lattice parameters calculated from XRD data by using the Rietveld refinement analysis are shown in Figures 3 respectively. All available reflections were fitted with the Gaussian distribution. Analysis of XRD patterns Figure 3 suggested that the lattice parameters for NiO obtained with dry temperature  $T_d = 350^\circ\text{C}$  are  $a = b = 3.255 \text{ \AA}$  and  $c = 5.218 \text{ \AA}$ . There is no significant difference in calculated lattice parameters as dry temperature is increased from 400°C. These results are slightly higher than the standard JCPDS parameter for bulk NiO,  $a = b = 3.2498 \text{ \AA}$  and  $c = 5.206 \text{ \AA}$ .

#### 3.5.1 Particle Size Calculation

From this study, considering the peak at degrees, average particle size has been estimated by using Debye Scherrer formula

Particle Size prediction by the Debye Scherrer formulae

$$D = 0.9\lambda / \beta \cos \theta$$

$$\lambda = 1.5406 \times 10^{-10} \text{ m}$$

$\beta$  = Full width at half maximum (radian)

Calculation of d-spacing

The value of d (the interplanar spacing between the atoms) is calculated using

Bragg's Law:  $2d \sin \theta = n\lambda$

$$\lambda$$

$$d = \frac{\lambda}{2 \sin \theta} \quad (n=1)$$

Wavelength  $\lambda = 1.5418 \text{ \AA}$

**Dislocation density ( $\delta$ ) is calculated with the crystalline size.**

$$\delta = \frac{1}{D^2}$$

**Table- 4 Simple peak indexing N1**

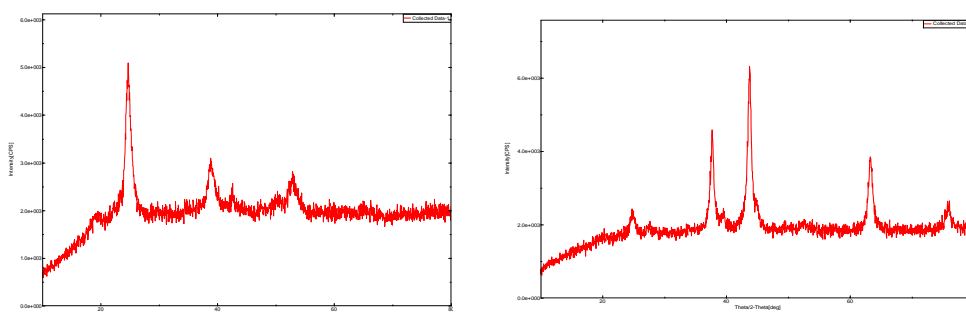
Peak position $2\theta$	$1000 \times \sin^2 \theta$	$1000 \times \sin^2 \theta / 40$	Reflection	Remarks
46.72	165.4	4.1	200	$2^2+0^2+0^2=2$
54.03	260	5.5	210	$2^2+1^2+1^2=5$
67.13	360	6.4	211	$3^2+0^2+0^2=6$

**Table - 5 The grain size of NiO Nanoparticles.**

S. No.	Compound	Size of the particle (D) nm	Dislocation density ( $\delta$ )
1	N 1	35.06	0.0173
2	N 2	40.64	0.0186

Moreover, a careful analysis of peak positions suggestive a small shifting in its value toward a lower  $2\theta$  with increasing dry temperature, indicating a presence of compressive strain in the samples. It is also shown that for the entire samples the reflection peaks become

sharper and the full width at half maximum (FWHM) are slightly decreased with increasing dry temperature. Using the Scherrer peak broadening method, the average crystallite sizes obtained are  $\sim 50$  nm.

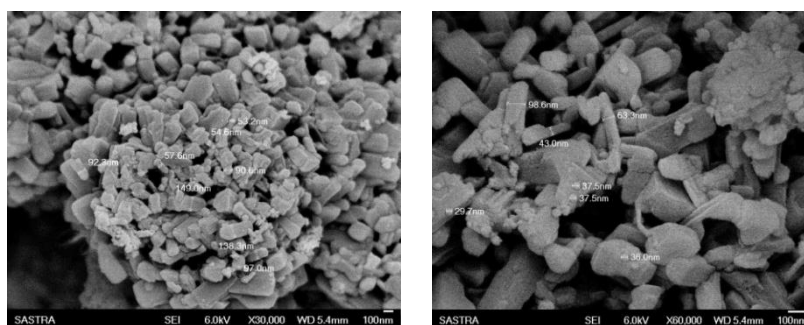


**Figure – 3 XRD graph for NiO nanoparticles N1 and N2**

### 3.6 Scanning Electron Microscope

The Morphology of synthesized NiO Nanoparticles was investigated by scanning electron microscopy (SEM) and elemental composition is analyzed by Energy dispersive X-ray spectroscopy (EDS). In **Figure 4** shows the SEM image of NiO synthesized from green method. NiO

nanoparticle synthesized from green method shows Spherical shape and the particles are highly agglomerated. As seen from the SEM images, green NiO nanoparticle has lower particle size. Particles were found to be around 100 nm, but were in agglomerated form.



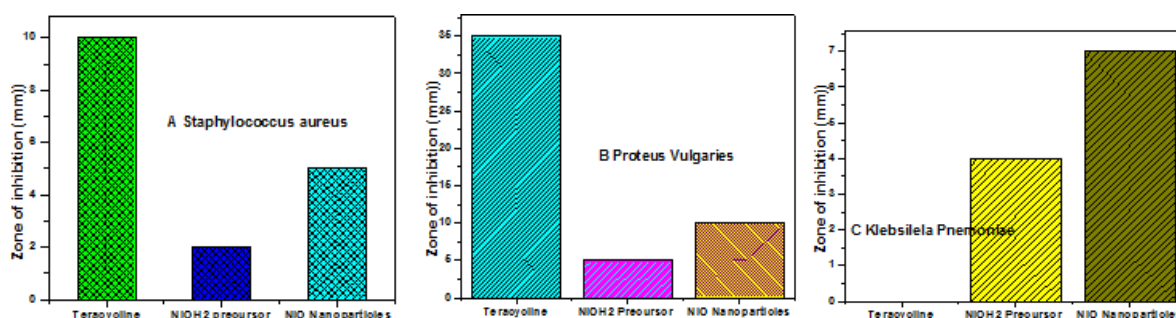
**Figure - 4 SEM image of NiO nanoparticles.**

### 3.7 Antibacterial activity of NiO Nanoparticles.

The antibacterial activity of NiO Nanoparticles was studied against a *Klebsiella* and *Staphylococcus aureus* bacterial pathogen. The selection of the organisms was based on their roles for causing infections such as diarrhea in both children and early weaned piglets. NiO nanoparticles at a concentration of 10 mg/ml showed

inhibitory effect against the growth of both *Klebsiella* and *Staphylococcus aureus* [28-30]. The clear zone of inhibition around the discs was the evidence of antibacterial activity, which is presented in Figure 5. Results showed that NiO Nanoparticles had good antibacterial activity against *Klebsiella* and *Staphylococcus aureus*.





**Figure 5(A-C) The diameter zone of inhibition (ZOI) of Drug, NiOH<sub>2</sub> Precursor and NiO nanoparticle impregnated disks in presence of *Staphylococcus aureus*, *Proteus Vulgaries* and *Klebsiella Pnemoniae* microorganisms.**

#### 4. CONCLUSIONS

In this work, NiO nanoparticles were bio synthesized using Asteracea Sirreu Sunrise Flower Extract. Average crystallite size of the crystals is 52 and 64 nm. The lattice constant of the NiO nanoparticles is larger than the bulk value. SEM analysis reveals that the nanoparticles were spherical in shape and Antimicrobial activity also found. NiO nanoparticles were prepared by simple Green method and characterized using UV, FT- IR, XRD and SEM with EDX. Green method has been found to be an easy, cheap, very fast and environmentally friendly compared to other chemical methods. The annealed sample shows decrease in agglomeration in their morphology. Antibacterial Studies It has been well acknowledged that NiO nanoparticles have good antibacterial activity. In the future, NiO nanoparticles could replace some antibiotic medicines used to combat human pathogenic microorganisms (bacteria), safe and cost effective in the Pharmaceutical industry.

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