


## Plant Extract Mediated Synthesis of Nickel Oxide Nanoparticles

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**Abstract:** The present study reports the successful synthesis of nickel oxide nanoparticles using *Vernonia amygdalina* plant leaf extracts as a chelating agent and nickel (II) chloride hexahydrate ( $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ) as precursor. The synthesized powder was gray black in color and annealed at 500 °C for 2 hours to obtain nickel oxide nanoparticles. Characterization techniques such as powder X-ray diffraction, scanning electron microscopy, and Fourier transform infrared spectroscopy were used to study the structure and morphology of the nanoparticles. Powder X-ray diffraction patterns revealed that nickel oxide nanoparticles with an average crystallite size of 17.86nm were synthesized. Scanning electron microscope images show that the nanoparticles have octahedral structure. Fourier transform infrared spectrophotometer analysis revealed that the strongest bond at  $1094.8\text{cm}^{-1}$  corresponds to stretching vibration mode of Ni-O nanoparticles.

**Keywords:** *Vernonia amygdalina*; NiO nanoparticles; plant extract mediated.

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### 1. Introduction

Nickel oxide (NiO) is a p-type wide band gap (3.6 to 4.9 eV) semiconductor with high chemical stability and electron transfer ability [1]. NiO is cubic structure and it is antiferromagnetic at a temperature of about 523 K [2]. NiO nanoparticles possess distinctive properties such as large surface area-to-volume ratios, low porosity, high dispersion rates, high photo-absorption, and low heat capacities [3]. These unique properties of NiO nanoparticles make viable and cost-effective surfaces suitable for different applications such as hydrogen storage [4, 5], photocatalytic degradation of organic dyes, pollutants from waste water and antimicrobial activity [6].

So far, various synthesis methods such as electrochemical reduction [5], chemical reduction, [7], sol gel [8] have been reported to synthesize NiO nanoparticles. Of late, the biomediated synthesis method has been proposed as a cost-effective, non-toxic and environmentally friendly alternative to chemical and physical methods [9-11]. In plant mediated synthesis of nanoparticles, extract of different parts of a plant is used as reducing agent and stabilizing agents [12, 13]. The nanoparticles produced by plant extracts are more stable and biocompatible in comparison with those produced by physical and chemical methods [11]. However, conditions such as calcinations time [14], temperature and reaction time [15, 16] which affects

the successful synthesis of NiO nanoparticles needs focused attention.

Recently, NiO nanoparticles have been synthesized using microorganisms, enzymes and plant extracts. For instance, Ezhilarasi et al. [17] used *Moringa Oleifera* to successfully synthesize NiO nanoparticles and investigated their biomedical application for cancer cell treatment. Nasser et al. [18] synthesized NiO nanoparticles using aqueous extract of *Tamarix serotina* and evaluated their catalytic activity and obtained cubic structure of NiO nanoparticles with average crystallite size between 10 to 14 nm. Ayesha Mariam, et al. [19] also reported the successful synthesis of pure and single phase NiO and Ni nanoparticles using leaves of *Azadirachta indica* and *Psidium guajava*. XRD patterns revealed that the obtained particles were pure cubic face centered with average crystallite size of 22 nm and 44 nm, respectively. In the present study, the leaves extract of *Vernonia amygdalina* (*V. amygdalina*) was employed as a surface stabilizing agent to synthesize nickel oxide nanoparticles, for the first time.

## 2. Materials and Methods

### 2.1. Materials.

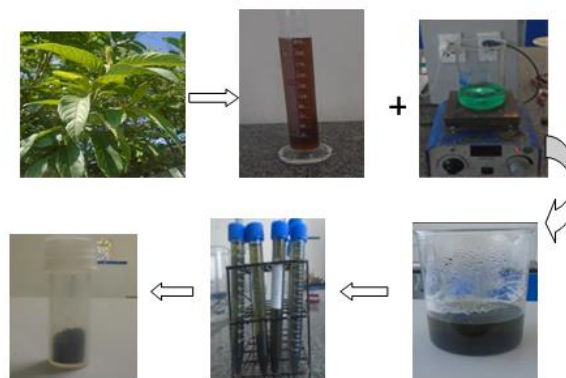
Nickel (II) chloride hexahydrate ( $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ) with purity of 99%, were purchased from Titan Biotech Ltd., India. Fresh and health leaves of *V. amygdalina* were collected from Debre Berhan University campus.

### 2.2. Methods.

The collected *V. amygdalina* leaves were thoroughly cleaned with running tap water to remove debris and other contaminants, followed by distilled water and air dried at room temperature for a week. The aqueous extract of the leaves was prepared by boiling the leaves with distilled water at about 60 °C for 50 minutes until the color of the aqueous solution changes to brown red. The extract was cooled to room temperature and filtered using Whatman No. 1 filter paper. Finally, the extract was stored in a refrigerator at 4 °C in order to be used for further experiments.

To prepare the precursor solution, 2.4g of nickel (II) oxide hexahydrate ( $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ) is mixed with 100ml distilled water and heated under magnetic stirring for ten minutes.

*V. amygdalina* is a small shrub which belongs to the *Asteraceae* family, a very popular plant that grows predominantly in the eastern and western parts of tropical Africa and is popularly called *bitter leaf* in English [20]. *V. amygdalina* grows in most parts of Ethiopia and known as ‘Grawa’ in Amharic [20]. The leaves of *V. amygdalina* have found relevance in traditional folk medicine as anti-helming, a laxative herb and an antimalarial [21] as they are known as quinine substitute [20, 22]. *Vernonia amygdalina* has a lot of therapeutic elements such as saponins, venomygdin and vernodaline; all of which have antibacterial properties and are useful in maintaining the health of other organs of the body like kidney and liver in traditional medicine [20]. Leaf decoctions are used to treat fever, malaria, diarrhoea, dysentery, hepatitis and cough, as a laxative and as a fertility inducer [21]. They are also used as a medicine for scabies, headache and stomach-ache, root extracts are also used as treatment against malaria and gastrointestinal disorder [23].



**Figure 1.** Synthesis process of NiO nanoparticles

To synthesize NiO nanoparticles, 50 ml of the precursor solution was mixed with 50 ml *V. amygdalina* aqueous extract in a 1:1 ratio. To achieve this, in every 3 minutes interval, 10 ml of the aqueous extract was slowly poured into the precursor solution until one can observe significant color change. The final mixture was stirred using magnetic stirrer for an hour under ambient conditions. After cooling down to room temperature, the resulting solution was washed three times, centrifuging each time at 3500 rpm with



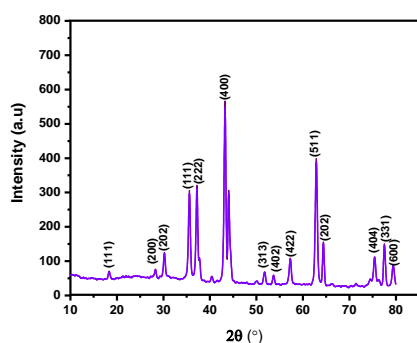
distilled water followed by ethanol to remove unwanted impurities. The resulting precipitate was collected and oven dried at 240 °C. Finally, the powder was calcined using air furnace for 2 hours at 500 °C to collect NiO nanopowder. After calcination, the NiO powder sample was carefully

placed in a clean sample holder for further characterizations of the nanoparticles. The overall synthesis process is shown schematically in **Figure 1** above.

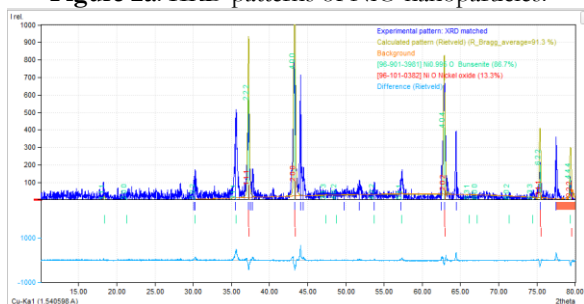
### 3. Results and Discussion

#### 3.1. X-Ray Diffraction (XRD).

For phase identification, XRD patterns were collected on an X-ray diffractometer (Adama Science and Technology University, Ethiopia) with 2 $\theta$  geometry, operating at CuK $\alpha$  radiation ( $\lambda=1.540598\text{\AA}$ ) at a voltage of 40kV and current of 30mA. The XRD patterns of all randomly oriented powder specimens were recorded in the 10° to 80° 2 $\theta$  range with a step size of 0.02 and a scan speed of 3°/min.



**Figure 2a.** XRD patterns of NiO nanoparticles.



**Figure 2b.** Rietveld refinement of NiO nanoparticles.

The XRD patterns for the prepared NiO nanoparticles are shown in **Figure 2a**. The observed peaks revealed that the obtained particles are cubic face centered with average crystallite sizes of 17.86nm. The prominent XRD diffraction peaks are indexed to 35.6°, 37.22° and 43.24° and the corresponding planes are (111), (222) and (400), respectively. The diffraction peaks indexed to a pure cubic phase of NiO with  $a = b = c = 8.3532\text{\AA}$ . Using Rietveld refinement, shown in **Figure 2b**, pure

NiO nanoparticles were successfully synthesized in the process.

The average crystallite size ‘D’ of the as-prepared NiO nanoparticles is calculated using Scherer equation:

$$D = \frac{0.9\lambda}{\beta \sin\theta} \quad (1)$$

Where ‘ $\lambda$ ’ is the wavelength of the incidence beam ( $\lambda=1.5406\text{\AA}$ ) and ‘ $\beta$ ’ is the full width at half its maximum, ‘ $\theta$ ’ is the Bragg’s diffraction angle.

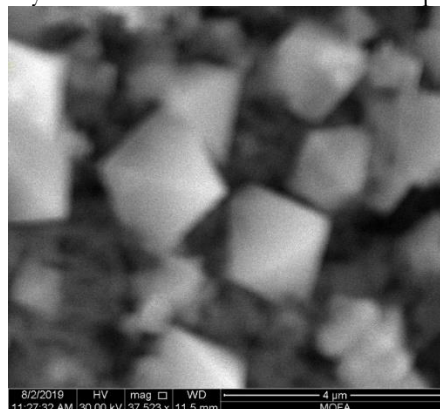
For face centered cubic crystal structure of NiO, the inter planar spacing ‘d’ is calculated using

$$d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}} \quad (2)$$

where ‘a’ represents the lattice parameter constant and ‘h’, ‘k’ and ‘l’ are the miller indices.

#### 3.2. Scanning electron microscopy (SEM).

**Figure 3**, below, shows SEM image of the as-synthesized NiO nanoparticles which revealed octahedral structure with cubic face centered geometry and this matches with bunsenite phase.

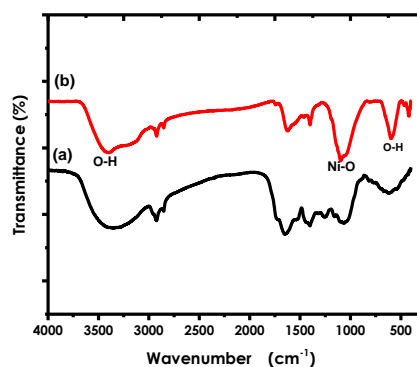


**Figure 3.** SEM images of NiO nanoparticles.

#### 3.3. Fourier transform infrared (FTIR) spectroscopy.

For FTIR analysis, pellets were prepared by mixing the synthesized NiO powder with KBr. The FTIR spectrum was carried out using spectrum 65 FT-IR (PerkinElmer) in the range of 4000-400cm<sup>-1</sup>. **Figure 4**, below, shows spectral peak were observed at 418.6cm<sup>-1</sup>, 596.02cm<sup>-1</sup>, 1094.8cm<sup>-1</sup>, 1402.2cm<sup>-1</sup>, 1627.05cm<sup>-1</sup>, and 3401.05cm<sup>-1</sup>. The

strong bond corresponding to Ni-O stretching vibration mode of NiO nanoparticles is observed at  $1094.8\text{cm}^{-1}$  [24-26]. The bonds at  $3401.05\text{cm}^{-1}$  and  $596.02\text{cm}^{-1}$  are characteristic of hydroxyl group (O-H) [25, 26], which may arise due to the adsorption of water molecules on to NiO surface while preparing the sample and exposure to atmospheric air [27].



**Figure 4.** FTIR spectra of (a) *V. amygdalina* plant leave extract, and (b) as-synthesized NiO nanoparticles.

#### 4. Conclusions

In conclusion, NiO nanoparticles were successfully synthesized using *V. amygdalina* plant leaves extract as chelating agent and nickel (II) chloride hexahydrate ( $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ) as a precursor. XRD patterns showed that the synthesized NiO nanoparticles have average crystallite size of 17.86 nm and exhibit bunsenite phase. From SEM images, it is clearly observed

that the synthesized NiO nanoparticles have octahedral structure. The Rietveld refinement also confirms that the synthesized NiO nanoparticles are cubic face centered crystal structure. From FTIR analysis the strongest bond corresponding to the Ni-O stretching vibration mode of NiO nanoparticles was observed at  $1094.8\text{cm}^{-1}$ .

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#### Conflicts of Interest

The authors declare no conflict of interest.

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