



Green Synthesis and Characterization of Nickel Oxide Nanoparticles Using *Plectranthus amboinicus* Leaf Extract

Abstract: The aim of this study is to synthesize the nickel nanoparticles of *P. amboinicus*, to identify them by spectroscopic techniques. After removal of the solvent from each extract by reduced pressure, the crude extracts were yielded. Methanolic extract was used for synthesis of nickel nanoparticles (s-NiONPs). The extract dissolved in distilled water was treated with nickel oxide solution to produce the s-NiONPs. The extensive spectroscopic study enabled the identification of synthesized s-NiONPs. The slight differences between extract and NiONPs in FTIR spectrum proved the formation of s-NiONPs. In that, some functional group such as hydroxyl in the extract was oxidized while reduction of nickel ions. The average particle size of s-NiONPs was measured as 71.6 nm by scanning electron microscope (SEM). Elemental analysis displayed the nickel percentage to be 66.12 %. X-ray diffraction analysis presented the maximum absorption of s-NiONPs at 486 nm. X-ray diffraction (XRD) pattern revealed that the crystal size of NiO nanoparticles (the value at 33.5 nm) were crystalline phase formation..

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Introduction

Green synthesis of nanoparticles using plant extracts is one of the cleanest, biocompatible, non-toxic, and eco-friendly methods for large-scale production of nanoparticles (Vinoth *et al.*, 2015). The main component present in plant extracts, namely polyol, acts as a chelating and capping agent for rapid biosynthesis of nanoparticles and stabilizes the formation of metallic nanoparticles. The majority of review supports that metal oxide nanoparticles such as silver, ferric, copper, gold, and nickel are synthesized via green synthesis route, with excellent structure/morphology and better physico-chemical properties (Varma *et al.*, 2011).

Biologically assisted synthesis of metal oxide nanoparticles plays a vital role in the field of nanomaterials. The function of environmentally benign materials such as plant

extract, fruit extracts, fungi and bacteria for the production metal oxide NPs considered as an eco-friendly approach. The advantage of method is based on the reaction between stoichiometric approximations. That means many researchers have practiced catalysis to face environmental problems and to eliminate hazardous chemicals from different sources since it is an effective green technology for water extraction and the complete elimination of toxic chemicals in the environment (Kaviyarasu *et al.*, 2017). Many studies publicized that using plant the extracts in synthesis of nanomaterial act as reducing agent and found to be more favourable due to low cost and good yield of NPs (Raja *et al.*, 2018). The scope of NPs applications is due to distinctive and attractive properties like magnetic, optical, chemical, electronic, mechanical, sensing, electrochemical (Abdullah *et al.*, 2021).

The phytochemical and bioactive compounds present in the plant extracts function as both natural reducing agents as well as capping agent which eliminates multiple steps and utilization of toxic, cost effective chemicals in the development of capped NPs (Angel ezhilarasu *et al.*, 2018). From the optical study shows that the energy band gaps of NiO direct allowed band gap is 3.47 eV by Ghougali *et al.*, 2016; Muhammad Imran Din and Aneela Rani, 2016; Saravanakumar *et al.*, 2019; Sharma *et al.*, 2015. The optical absorption spectra of NiO nanoparticles have been reported that the band gap value of NiO films was found from 3.64 eV to 3.86 eV. This shift predicts that there is an increase in band gap value ($E_g = 3.86$ eV), which

is due to the reduction in particle size ($D = 9.72$ nm) (Nurul Nadia Mohd Zorkipli *et al.*, 2016).

Plectranthus amboinicus (Lour) Spreng belongs to family Lamiaceae and is known as country borage in English. It is a large succulent aromatic perennial herb, shrubby below, hispidly villous or tomentose. Upon literature review it was found that the plant contains butylanisole, β -caryophyllene, quercetin etc. Many pharmacological properties have been reported including urolithiasis (Chatterjee and Satyesh (2001), antiepileptic, antitumor and antimutagenic, neuropharmacological, radioprotective effect, antioxidant, antimicrobial, antibacterial, antifungal properties. Our present study determined the *Plectranthus amboinicus* leaves extracts moderate nickel nanoparticles synthesis. The synthesis NiONPs was confirmed by Fourier transform infra-red spectroscopy (FTIR), the scanning electron microscope (SEM) and X-ray diffraction analysis.

Material and Methods

Collection and identification of the plant material

The fresh leaves of *Plectranthus amboinicus* was collected from Gudiyatham, Tamil Nadu. The sample was authentically identified from Department of Botany, K. K. Government Arts College, Tiruvannamalai. The leaves were thoroughly rinsed with tap water and ensured that it was devoid of contaminants. The leaves was air dried at room temperature and pulverized to fine powder and stored in airtight container at room temperature for further analysis. Materials used for the synthesis process were Nickel oxalate dihydrate ($\text{Ni(OH)} \cdot 2\text{H}_2\text{O}$) and Sodium Hydroxide (NaOH).

Extraction procedure

Air dried plant material (200 g) was extracted with hexane for 3 days. After filtration, the solvent was removed by reduced pressure to yield the crude hexane extract. The solid material was re-extracted with dichloromethane, ethyl acetate and methanol sequentially to yield the crude extracts. The methanol extract was used for synthesis of s-NiONPs.

Synthesis of nickel nanoparticles

The methanol extract of *Plectranthus amboinicus* (1.0 g) was dissolved in distilled water (300 mL). The nickel oxide solution (0.024 M, 300 mL) was added to the extract solution slowly. The final reaction mixture was mixed at 55°C for 2 h. The colour change from yellow to brown showed the establishment of s-NiONPs. The mixture was cooled to room temperature then, applied to centrifugation at 15,000 rpm for 10 min, washed with distilled water.

Synthesis of Nickel Oxide

In the typical procedure, 50 ml of 3N solution of $\text{Ni(OH)} \cdot 2\text{H}_2\text{O}$ was made in distilled water and mixed NaOH and the same procedure for synthesized NiO nanoparticles using *P. amboinicus* leaves extract instead of NaOH for green synthesis method. The solution was maintained at constant stirring for 3 hours under controlled manner. The bluish green precipitate solution was obtained, and it was rinsed with de-ionized water until the pH becomes neutral. The precipitate was dehydrated in a hot air oven at 80°C . Finally, bluish green powder was crushed and then calcined at 400°C for 1 hour. Hence, we collect NiO nanoparticles for further characterization study.

The phase analysis, functional groups and optical absorption properties of pure NiO nanoparticles were characterized by FTIR, SEM-EDX and X-ray diffraction analysis.

Results and Discussion

FTIR Analysis:

FTIR spectrum presented the functional groups responsible for the nickel-oxide reduction and stabilization of s-NiONPs. The absorption differences in the FTIR spectrum of s-NiONPs and *Plectranthus amboinicus* leaf extract confirmed the formation of s-NiONPs. These differences are caused by the oxidation of functional groups such as hydroxyl while reduction of Nickel-oxide. The characteristic vibration signals of extract and s-NiONPs for hydroxyl groups appeared at 2322.29, 648.03, 594.08, 547.78, 447.49, 416.62cm^{-1} , respectively (Table-1). Hence, nickel-oxide could be reduced to NiO by plant metabolites such as proteins and flavonoids. Moreover, s-NiONPs was capped and stabilized by functional groups (Figure-1).

Table-1. Determination of functional groups in *Plectranthus amboinicus* leaves

S. No	Frequency, cm ⁻¹	Functional group
1.	2322.29	Alkanes C-H Stretching
2.	648.08	NiO Stretching
3.	594.08	NiO Stretching
4.	547.78	NiO Stretching
5.	447.49	NiO Stretching
6.	416.62	NiO Stretching

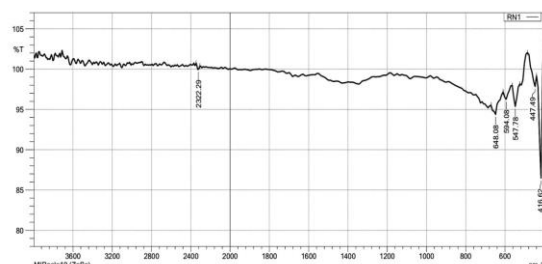
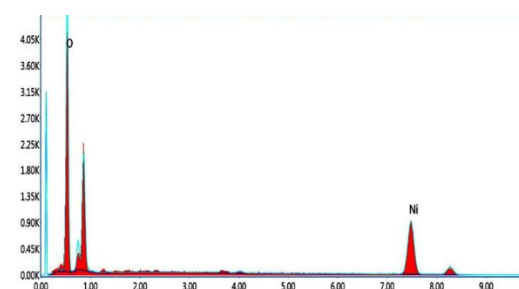
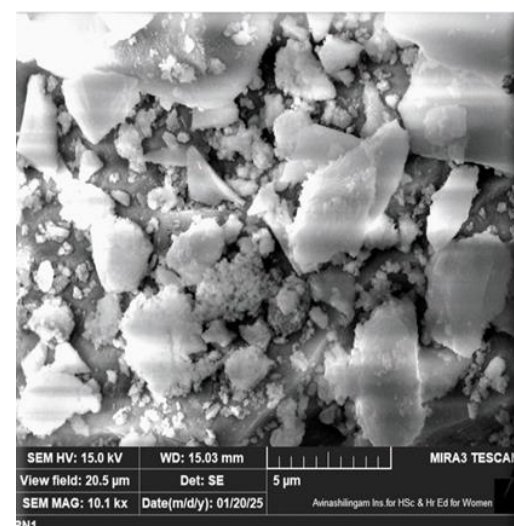
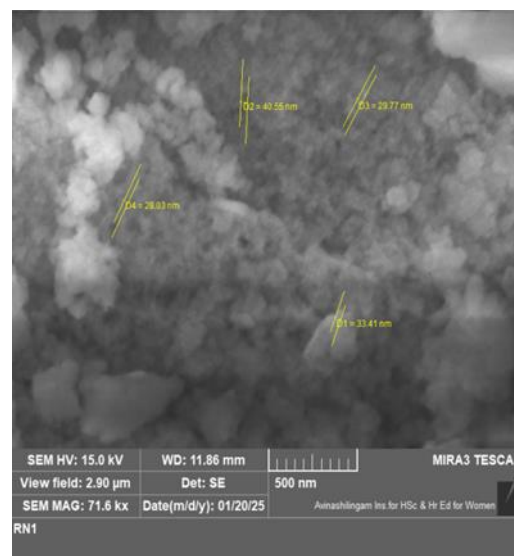


Fig. 1. FT-IR spectrum of *Plectranthus amboinicus* leaves

Scanning electron microscope (SEM) and energy-dispersive x-ray spectroscopy (EDX) analysis.

The scanning electron microscope (SEM) presented the surface morphology and size of s-NiONPs. The energy dispersive analysis of x-rays (EDX) showed the presence of s-NiO₂NPs. Furthermore, remarkable intense peak of s-NiO₂NPs in EDX spectrum at around 7.2 and 7.6 keV confirmed the synthesis of s-NiNPs (Figure-2). SEM analysis of synthesized s-NiONPs was spherical shaped and measured as an average size of 71.6 nm. Moreover, elemental analysis results indicated that the nickel percentage to be 66.12 %.

The SEM observations of the NiO nanoparticles make known that the shape of the Ni particles is nearly spherical and the circulation of the particles (Huazhi wang *et al.*, 2008). For this rationale it may be implicit that the tentative circumstances pull off the mono-disparity and consistent profile (Khalaji *et al.*, 2014; Zahariev *et al.*, 2016). And it indicates that the Co crystals are formed by aggregation of smaller crystallites during the synthesis process and the morphology of NiO and GS-NiO nanoparticles is very uniform.



Element	Weight %	Atomic %	Error %
C K	5.03	12.83	12.31
O K	26.60	50.94	7.17
Ca K	2.25	1.72	7.90
Ni K	66.12	34.50	3.66

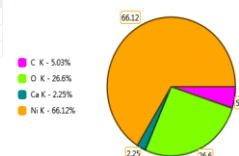


Figure-2. SEM-EDX spectrum and elemental analysis of s-NiONPs

X-ray diffraction analysis

The purpose of the X-ray diffraction analysis was to learn more about the crystalline makeup of the produced nanoparticle. The distinctive peaks seen in the XRD scan further verified and corroborated the biosynthesized nickel nanostructure created by using *P. amboinicus* leaf extract. The intense peaks in the entire spectrum of 2 values ranging from 20 to 80 are visible in the XRD spectrum of the NiO biophytum. The broadening of peaks indicates the formation of nanoparticle at 33.5 nm. Thus, XRD pattern clearly shows that the synthesized s-NiONPs was crystalline in nature (Figure-3). The crystallite size could be calculated by measuring the half-maximum value of the X-ray diffraction peak (FMHM) using the Debye-Scherrer formula (Atak and Skun, 2017).

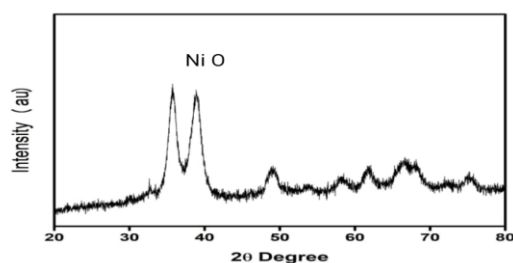


Figure-3. XRD Spectrum of NiO Nanoparticle.

Conclusion

Nickel oxide nanoparticles successfully synthesized using *P. amboinicus* leaves as reducing and capping agents. Nickel oxalate dehydrates was used as source for the synthesis of NiO nanoparticles. The synthesizing process is green, eco-friendly and sustainable in nature. The technique produced the highest yield possible given the starting ingredients used. Spectral characterization of the nickel oxide nanoparticles is confirmed by FTIR, SEM-EDX and XRD analysis.

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