

Green Synthesis And Characterization Of Nickel Oxide Nanoparticles

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Abstract

An Contemporary affirm of explore on the green synthesis of metal oxide nanoparticles and their improvement over chemical synthesis routine. In this method, non-hazardous safe reagents that are eco-friendly and biosafe are used. Consequently, an attempt is to prepare the Nickel Oxide (NiO) nanoparticles via Chemical and Green synthesis method. To Synthesize the pure NiO and GS-NiO nanoparticle with reducing agent of *Plectranthus Amboinicus* leaf extract used and calcinated at 400°C in a controlled manner. To study the phase nature, functional groups and Optical energy band gap of the synthesized pure NiO and GS-NiO nano particles by using XRD, FT-IR and DRS-UV.

Keywords: *NiO, Plectranthus Amboinicus leaf, XRD, FTIR, UV-DRS, SEM-EDAX.*

1.1 Introduction

A facile way of Green synthesis is a promising area in the pasture of nanoworld and provides economic and environmental benefits. Know-how to be in command of crystallization is an essential constraint for the synthesis, purification, and application of materials in various industrial fields. In particular, Nickel oxide (NiO) is one of the the majority expensive resources

in the crum of its electronic and optical properties, which are tough subjective by the various morphologies, crystal sizes, dimensions, and aspect ratios.

Nanoscience, the stage imperative in our universe, also it is considered a next planet in research field. Numerous researchers cover green synthesis routes for the preparation of metal oxide nanoparticles using unique plant. In few decades, nickel nanomaterials have been synthesized in various forms like nanotubes, nanorods, hollow spheres, nanobelts, nanoprisms, and hexagonal flakes. Synthesis of nickel oxide nanoparticles using pomegranate peels and fungal biomass are an alternative to chemical synthesis.

From the optical study shows that the energy band gaps of NiO direct allowed band gap is 3.47 eV [M. Ghougali et al., 2016, Muhammad Imran Din and Aneela Rani, 2016, A.K 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].

This investigations have not yet reported that the synthesise of NiO nanoparticles using Plectranthus Amboinicus leaf broth. In this Green synthesis method are requisite to circumvent the making of superfluous or detrimental by-product during the upsurge of dependable, sustainable, and eco-friendly synthesis procedures.

2. Materials and Methods

2.1 Materials

Materials used for the synthesis process were Nickel Oxalate dihydrate ($\text{Ni}(\text{OH}) \cdot 2\text{H}_2\text{O}$), Sodium Hydroxide (NaOH) and Plectranthus Amboinicus leaves.

2.2 Characterization Technique

In the present study the samples were characterized using the following analytical techniques XRD, FTIR, DRS-UV and SEM-EDAX. X-ray diffraction (XRD) study were recorded from

Alagappa University, Karaikudi, Tamil Nadu using XPERT- PRO diffractometer with Cu anode materials ($K\text{-Alpha} = 1.5406 \text{ \AA}$). and Fourier Transform Infrared Spectroscopy (FTIR) & SEM-EDAX were confirmed through Kalasalingam University, Krishnankovil. Ultraviolet Visible-Diffuse Reflectance Spectroscopy (UV-DRS) Spectra were recorded in VOC college, Tuticorin.

3. Experimental Procedure

3.1 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 GS-NiO nanoparticles using *Plectranthus 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-ionised 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 and GS-NiO nanoparticles for further characterization study.

The phase analysis, functional groups and optical absorption properties of pure NiO and GS-NiO nanoparticles were characterized by XRD, FTIR, DRS-UV and SEM-EDAX techniques.

4. Results and Discussion

4.1. X-Ray Diffraction (XRD) Study

From the figure 4.1 demonstrates the XRD profiles of the NiO and GS-NiO nanoparticle prepared without any surfactant. The diffraction peaks were coincide with JCPDS card no: 471049, and the observed patterns can be explicitly recognized to the existence of cubic crystallites. Good crystallinity of NiO is received by the increase of crystallinity and its size can be effectively achieved after annealing treatment [Muyang Shi et al., 2021]. The crystal size of

NiO and GSNiO nanoparticles was calculated from FWHM and the value is 40.85 nm and 30.54 nm with respectively.

That the green synthesis of nanoparticles which reduces the particle size and enhances the strength counterpart. The peak intensity markedly increases with an increase of calcination process. The incorporated of NiO and GS-NiO nanoparticles and the temperature is the vital part crystalline phase formation.

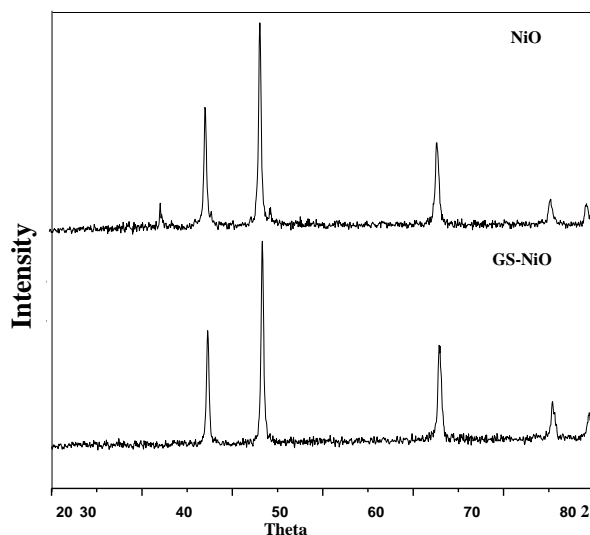


Figure 4.1. XRD Spectrum of NiO and GS-NiO Nanoparticle

4.1. Fourier Transformation Infrared Spectroscopy (FT-IR) Study

FTIR analysis reported the qualitative and quantitative elements mode of vibrations. The main functional groups of NiO and GS-NiO nanoparticles are identified using available literature and their band assignments are reported. In the Fig. 4.2 shows that the spectra of NiO and GSNiO nanoparticles were calcination at 400°C.

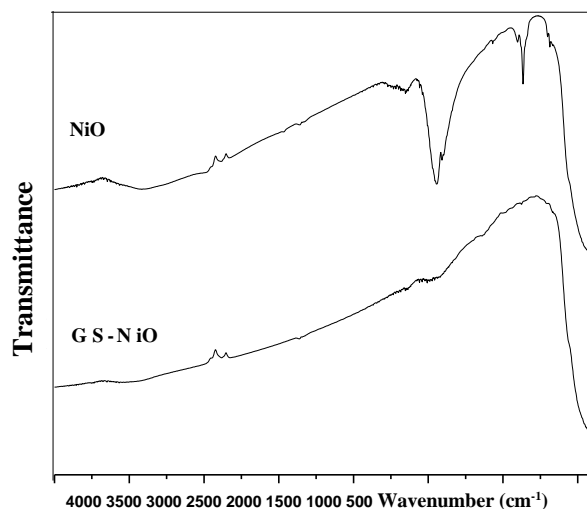


Figure 4.2. FTIR spectra of NiO and GS-NiO Nanoparticles

The broad peak centered at 3441 cm^{-1} is attributed O-H stretching vibration band and the weak band near 1406 cm^{-1} is assigned to O-C=O carboxyl groups in vibrations mode. These observations provide the confirmation to the cause of hydration in the formation. The peak at 1504 cm^{-1} serrated the carboxyl groups of the green synthesis nanoparticle. The wavenumber shift is higher in GS-NiO nanoparticle than the NiO nanoparticle. The peak value at 662 cm^{-1} in NiO and the higher shift occurs at 682 cm^{-1} in GS-NiO particles.

The peak at 682 cm^{-1} indicates the nanocrystalline nature of NiO wagging vibration mode. The band around at 651 cm^{-1} corresponds to in-plane bending mode of vibration of NiO and also the peak at 575 cm^{-1} indicated that the bending vibration of NiO particles [Omolola E. Fayemi et al., 2016, M.K. Mahmoudabad et al., 2011, Nisha J Tharayila et al, 2008].

4.3. Diffuse Reflectance Spectroscopy (DRS-UV-Vis) Study

The Optical study is recorded on DRS UV-Vis absorption spectrophotometer to facilitate determines the energy bandgap value of NiO and GS-NiO. From the Fig.4.3. shows that the hint of the NiO and GS-NiO nanoparticle calcinated at 400°C . The peak absorbance of photon energy in the visible region ranging from 320 to 400 nm indicates the good fascination nature of NiO and GS-NiO nanoparticles. An energy band gap and the transition of both samples were firm by Kubelka-Munk function

$$F(R) = \frac{(1-R)^2}{2R} \quad \dots \text{ I}$$

An indirect band gap of NiO and GS-NiO was calculated by plotting $[F(R) \cdot hv]$ vs the energy of photon (eV) is offered in the fig. 4.4. From the tauc plot was extrapolated to get the energy band gap (E_g) value is 3.5 eV and 3.4 eV with respectively.

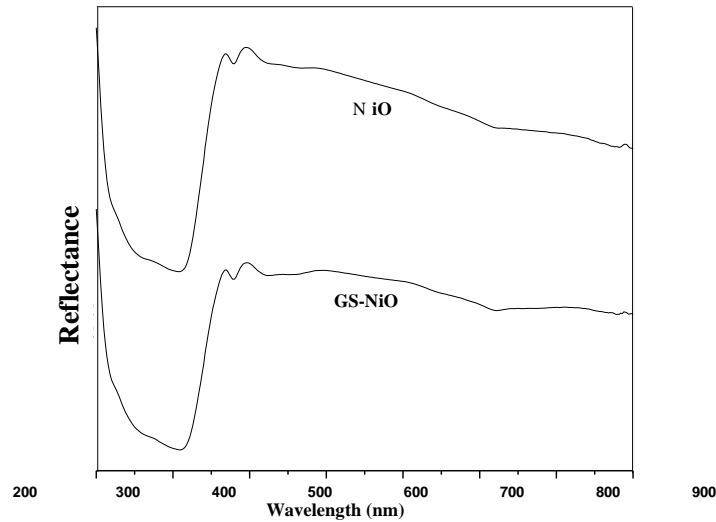


Figure 4.3. DRS Spectrum of NiO and GS-NiO Nanoparticles

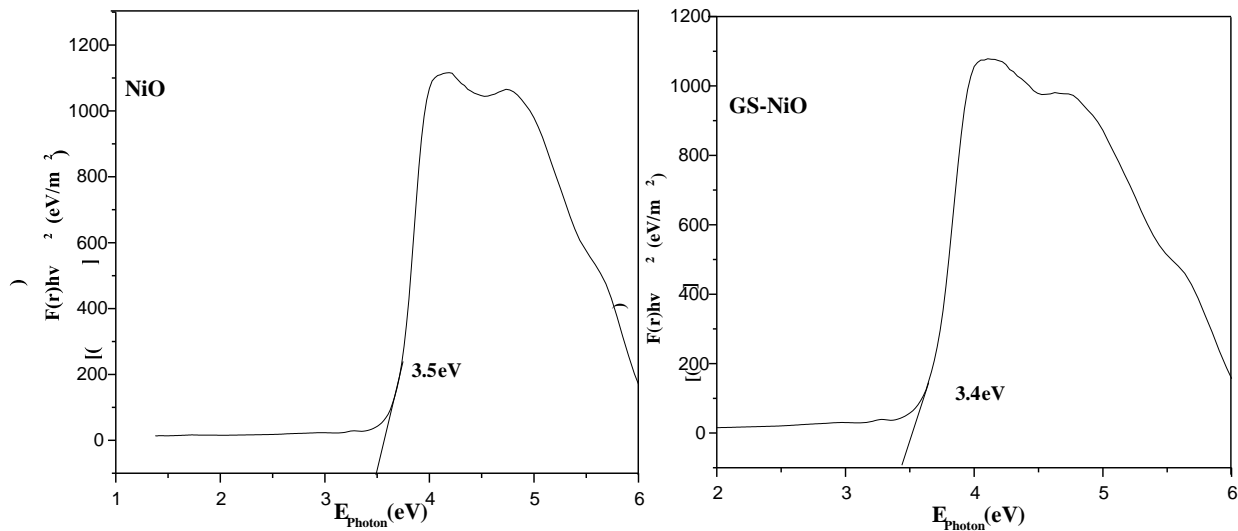


Figure 4.4. Tau Plot of NiO and GS-NiO Nanoparticles

Wavelength of absorption spectra increases or above the cut-off region the absorption is constant, and here it shows the transparent nature of the nickel oxide nanoparticles. An

absorption spectrum of NiO nanoparticles monitored at 354 nm. [Amrut. S. Lanjeet al, 2010]. The absorbance wave length values is 344 nm and it very closely matched with the reported values[Ansilin et al, 2016].

4.4 Scanning Electron Microscopy (SEM) Study

The SEM micrographs with EDAX reports the NiO and GS-NiO nanoparticles are shown in Fig. 4.5. The surface morphology of the samples was recorded with low and high magnification. The weight percentage of elements in the samples is reported through EDAX spectrum. The surface texture of the nickel nanoparticles are within and morphology study has found out the synthesized nickel nanoparticles are moderately spherical with thorn like structure.

The SEM observations of the NiO and GS-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 will pull off the mono-disparity and consistent profile [A.D. Khalaji et al, 2014 & Ivan Z.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 much uniform.

Various shapes are seen in the morphological texture of the NiO and GS-NiO nanoparticles in with various magnifications. The particles are in flower shape at the high magnification of 50.00KX. The flakes structure and hexagonal shape of the NiO and GS-NiO particles are noticed with 200 nm approximate size were also observed. Even homogeneous spherical partiles with porous structure are seen in low magnification.

Visualized images indicating the variation in particle sizes and the average size estimated was 200 nm NiO and GS-NiO nanoparticles with respectively. The natal molecules could probably act upon twofold functions of pattern and stabilization of nanoparticles in the combination method.

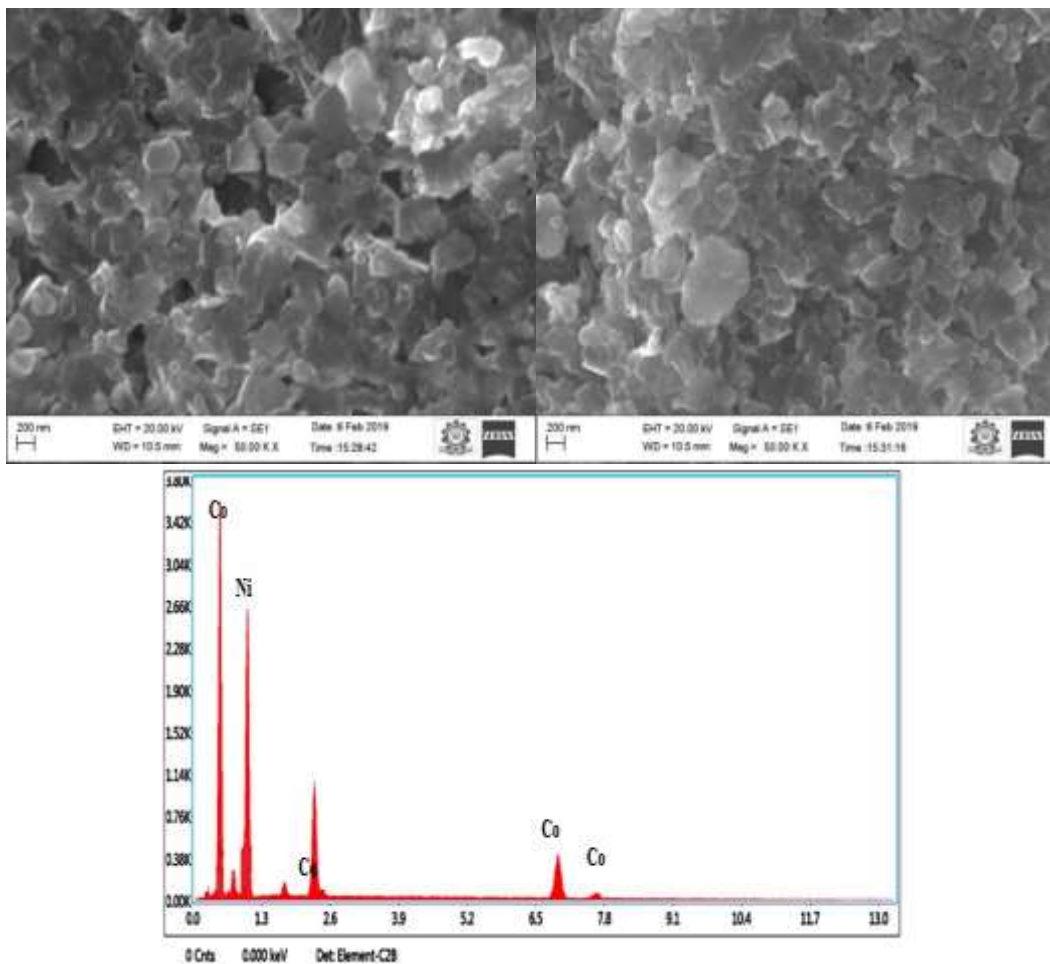


Figure 4.5 SEM-EDAX Micrographs of Ni-Co Nanoparticles

4.5 Energy dispersive X-ray (EDAX) analysis

From the report confirms that NiO and GS-NiO was present approximately 95% (by wt.) with oxide composition [R. Manivann et al, 2015]. The texture surface of all images shows that the particles were identical and sphere-shaped and also particles were in agglomerated condition.

5. Conclusion

From this investigation, we conclude that the NiO and GS-NiO nanoparticles have been synthesized via Chemical and Green synthesis method with controlled stoichiometry ratio. The cubic formation was identified for green and chemically synthesised NiO nanoparticles in the course of XRD study. Due to the formation of well crystalline peaks estimated the particle size. The average particle size are 40.85 and 30.54 nm of NiO and GS-NiO with respectively. The strong peak at 682 cm^{-1} is endorsed to the Ni-O stretching tremor form and 430 and 410 cm^{-1}

represents the bending mode of Ni-O nanoparticle. The outer shell of a sharp band at 450 cm^{-1} is confirms the synthesis of NiO and also it is the distinctive inclusion band for the Ni–O stretching vibration. The DRS spectra of both NiO nanoparticles reflectivity were present in the significant region. The peak absorbance of photon in the visible region ranging from 354 to 344 nm of NiO and GS-NiO, which indicates the GS-NiO particle is optimum than NiO particles. The morphology study shows that the hexagonal shape of the Ni and GS-NiO particles are noticed with 200 nm approximate size were also observed. All the results well expected the synthesis technique be extended to prepare metal oxide nanostructures. The Optimum results have GS-NiO nanoparticle than the NiO particles.

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Conflicts of Interest:

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

References:

- [1] M. Ghougali, O. Belahssen, A. Chala, J.Nano-Electron. Phys. 8 No 4(2), 04059 (2016).
- [2] Muhammad Imran Din and Aneela Rani, International Journal of Analytical Chemistry, Article ID 3512145, (2016).
- [3] Abhishek Kumar Sharma, Sameerah Desnavi, Charu Dixit , Utkarshaa Varshney, and Ankur Sharma, International Journal of Chemical Engineering and Applications, Vol. 6, No. 3, (2015).

- [4] Nurul Nadia Mohd Zorkipli , Noor Haida Mohd Kaus , Ahmad Azmin Mohamad, *Procedia Chemistry* 19, 626 – 63, (2016).
- [5] Muiyung Shi, , Tian Qiu, Biao Tang, Guanguang Zhang, , Rihui Yao , Wei Xu, Junlong Chen, Xiao Fu, Honglong Ning, and Junbiao Peng, *Micromachines*, 12, 80, (2021). (in Chinese)
- [6] Omolola E. Fayemi, Abolanle S. Adekunle and Eno E. Ebenso, *Journal of Nanomaterials*, Article ID 9614897, 12, (2016).
- [7] M.K. Mahmoudabad, M. M. Kashani Motlagh., *International Journal of the Physical Sciences* Vol. 6(6), pp. 1471-1476, (2011).
- [8] Nisha J Tharayila, R. Raveendran, Alexander Varghese Vaidyan and P.G. Chithra., *Indian Journal of Engineering & Materials Sciences* Vol. 15, pp. 489-496, (2008).
- [9] Amrut. S. Lanje, Satish J. Sharma, Ramchandara B. Pode, Raghmani S. Ningthoujam, *Advances in Applied Science Research*, 1 (2): 36-40, (2010).
- [10] Ansilin. S, J. Kavya Nair, C. Aswathy, V. Rama, J. Peter, J. Jeyachynthaya Persis, *Journal of Nanoscience and Technology* 2(5), 221–223 222, (2016).
- [11] Huazhi Wang, Xinli Lou, Jie Zhang, Jiangong Li, *Bull. Mater. Sci.*, Vol. 31(1), pp. 97–100, (2008). (in Chinese)
- [12] A.D. Khalaji, M. Yarestani, A. Rohini, D.Das, *Journal of Sciences, Islamic Republic of Iran* 25(4): 339- 343 (2014).
- [13] Ivan Zahariev, Mehmet Piskin, Emre Karaduman, Dimka Ivanova, Ivania Markova, Ludmil Fachikov, *Journal of Chemical Technology and Metallurgy*, 52(5), 916-928, (2017).
- [14] Shanaj BR and John XR, *J. Theor. Comput. Sci.*, 3-2, (2016).