



SACRED HEART RESEARCH PUBLICATIONS

# Journal of Functional Materials and Biomolecules

Journal homepage: [www.shcpub.edu.in](http://www.shcpub.edu.in)



ISSN: 2456-9429

## INVESTIGATION ON THE SYNTHESIS AND CHARACTERIZATION OF NiO NANOPARTICLES

G. Jayakumar<sup>1\*</sup>, A. Albert Irudayaraj<sup>1</sup>, A. Dhayal Raj<sup>1</sup>, S. Savitha<sup>1</sup>

Received on 28 November 2023, accepted on 2 December 2023,

Published online on December 2023

### Abstract

The NiO NPs are synthesized by chemical precipitation route at different calcination temperatures 300°C and 400°C. The prepared samples are subjected to powder XRD, SEM, EDS, FTIR and PL analysis. The powder XRD study shows that both NiO NPs have FCC structure. The crystallite size of NiO NPs increases with increase in the calcination temperature. The peaks at 575 cm<sup>-1</sup> and 424 cm<sup>-1</sup> in FTIR spectra of both the samples are due to the Ni-O stretching vibrations. The SEM analysis reveals that the synthesized NiO NPs are spherical in shape. The average particle size increases with increase in the calcination temperature. The EDS analysis confirms the presence of Ni and O in the synthesized NiO NPs calcinated at 300°C and 400°C. The PL analysis shows that the NiO NPs, calcinated at 300°C and at 400°C, exhibited red emission peaks at 616 nm and 612 nm respectively. The intensity of the emission peaks increases with increasing calcination temperature.

**Keywords:** NiO, FCC, stretching vibrations, particle size, red emission

### 1. Introduction

Nanotechnology is an advanced technology, which deals with the synthesis, processing of nanoparticles and their applications [1-3]. The properties of nanoparticles are different from that of bulk materials in a significant way [4]. The nickel oxide (NiO) is a very important metal oxide functional material. It has FCC structure. It finds applications in catalysis, battery electrodes, electro chromic films, fuel cells, organic light emitting diodes, thermoelectric materials, gas sensors, and magnetic materials [5-7]. Among the various chemical methods, hydrothermal method is one of the most widely used technique for the preparation of NiO NPs which can easily be obtained through hydrothermal treatment of peptized precipitates. The hydrothermal method can be useful to control grain size, particle morphology, crystalline phase and surface chemistry through regulation of the solution composition, reaction temperature, pressure, solvent properties, additives and aging time [8-12]. In the present work, the NiO NPs have been synthesized by chemical precipitation method at different calcination temperatures and the effect of

calcination temperature on the properties of Nickel oxide nanoparticles have been studied by Powder XRD, SEM, EDS, FTIR, and PL analysis and the results have been discussed.

### 2. Experimental Method

#### 2.1 Materials

The following materials were used for the preparation of NiO NPs, Nickel chloride hexahydrate [NiCl<sub>2</sub> · 6H<sub>2</sub>O], Potassium hydroxide [KOH] pellets, Polyethylene glycol [PEG], Ethanol and Acetone with 99.99% analytic grade from Merck, Mumbai. Double distilled water was used for the preparation of all the solutions.

#### 2.2 Synthesis of NiO NPs

For the preparation of NiO NPs, 0.1M NiCl<sub>2</sub> · 6H<sub>2</sub>O solution and 0.2 M KOH solution were prepared. The KOH solution was slowly added into the precursor solution. 10 ml of PEG, a surfactant, was added into the solution and it was stirred for 2 hrs. The resultant solution was placed without disturbance for few hours. The obtained precipitate was washed several times by double distilled water and ethanol. Then, it was dried at 80°C for 6 hrs and calcinated at 300°C for 2 hrs. In order to study the effect of calcination temperature on the properties of NiO NPs, the dried sample was calcinated at 300°C and 400°C for 2 hrs.

#### 2.3 Characterization techniques

The structure and crystallinity of the NiO NPs are identified by Rigaku powder X-ray Diffractometer using Cu-K $\alpha$  radiation as the X-ray source. The morphology and particle size of the samples are examined by A FEI Quanta FEG 200F electron microscope coupled with energy dispersive x-ray spectrometer (EDS). The Perkin Elmer Spectrum – II Fourier Transform infrared spectra of the samples are recorded in the range 4000 cm<sup>-1</sup> - 400 cm<sup>-1</sup> using spectrometer. The photoluminescence spectra of the

\*Corresponding author: e-mail: [jayakumar@shcpt.edu](mailto:jayakumar@shcpt.edu)

<sup>1</sup> PG & Department of Physics, Sacred Heart College (Autonomous), Tirupattur - 635 601, Tamilnadu, India.

samples are recorded over the wavelength 400nm to 800nm by Perkin Elmer LS-45 photometer.

### 3. Results and Discussion

#### 3.1 Powder XRD analysis

The Powder XRD patterns of the NiO samples calcinated at (a) 300°C and (b) 400°C are shown in figure 1. The peaks are indexed with the help of the standard JCPDS file No: 78-0423. The peak positions appearing at  $2\theta \approx 37.27, 43.31, 62.87, 75.56,$  and  $79.49$  are indexed to (111), (200), (220), (311) and (222) respectively. Both the NiO samples calcinated at (a) 300°C and (b) 400°C have Face centered cubic structure with lattice parameters  $a = b = c = 4.179 \text{ \AA}$ . The diffraction patterns indicate the formation of NiO NPs with high purity. The crystallite size of NiO NPs calcinated at (a) 300°C and (b) 400°C was estimated using Scherrer equation. The estimated average crystallite size of NiO NPs calcinated at (a) 300°C and (b) 400°C was found to be 12.65 nm and 17.54 nm respectively. Thus, it is found that the average crystallite size increases with increasing calcination temperature. These results are in agreement with the observations reported by Ravi Kant sharma et al. [13] Mohammad Reza kalaie et al. [14], Karthick et al. [15] and M. M. Kashani motlagh et al. [16].

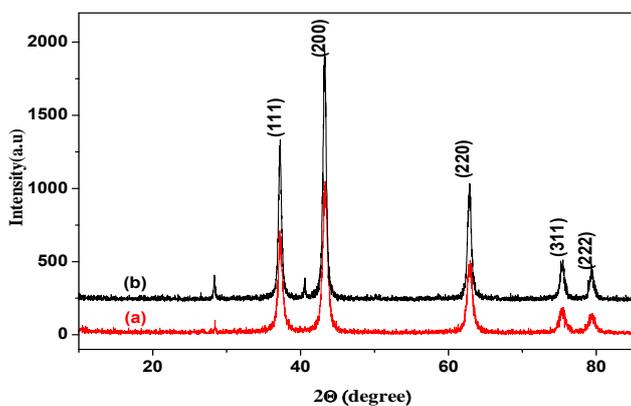


Fig. 1 Powder XRD pattern of the NiO NPs calcinated at (a) 300° C and (b) 400° C

#### 3.2 FTIR analysis

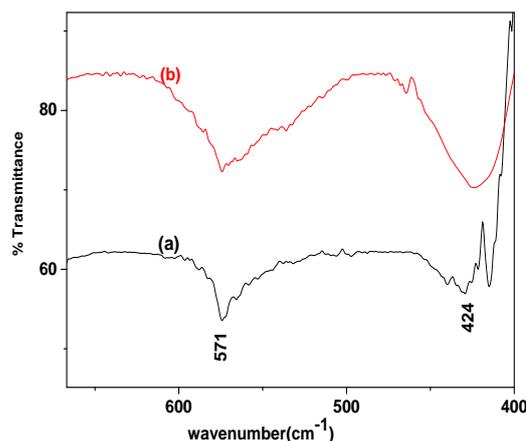
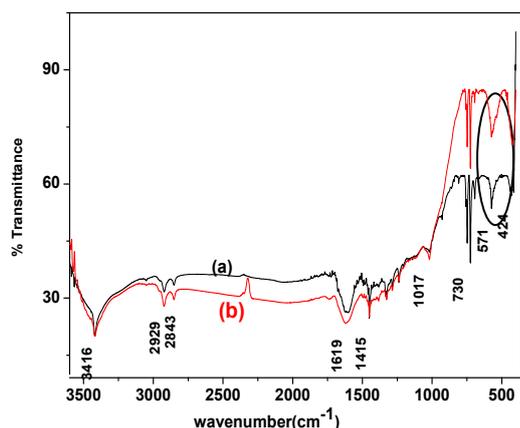


Fig. 2 FTIR spectra of the NiO NPs calcinated at (a) 300° C and (b) 400° C

The FTIR spectra of the NiO NPs calcinated at (a) 300°C and (b) 400°C are shown in figure 2. The peaks at 575  $\text{cm}^{-1}$  and 424  $\text{cm}^{-1}$  are due to the Ni-O Stretching Vibration [17] and it confirms the presence of NiO in the prepared samples. The peaks at 1017  $\text{cm}^{-1}$ , 2929  $\text{cm}^{-1}$ , 2843  $\text{cm}^{-1}$  and 3416  $\text{cm}^{-1}$  are due to moisture absorbed by KBr from the atmosphere.

#### 3.3 SEM analysis

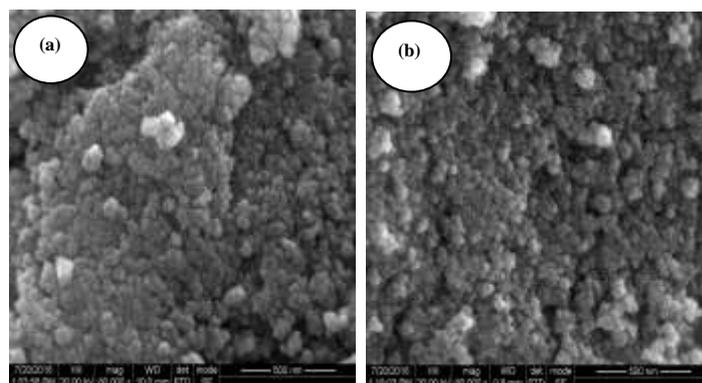


Fig. 3 SEM images of the NiO NPs calcinated at (a) 300° C and (b) 400° C

The SEM images of NiO NPs calcinated at (a) 300°C and (b) 400°C are shown in figure 3. The SEM images show that the particles are spherical in shape and slightly agglomerated. The average particle sizes are 24.84 nm and 27.36 nm for NiO NPs calcinated at 300°C and 400°C respectively. The SEM image clearly reveals that the average particle size of NiO NPs increases as the calcination temperature increases [18].

#### 3.4 EDS analysis

The EDS spectra of NiO NPs calcinated at (a) 300°C and (b) 400°C are shown in figure 4. The EDX result confirms the

presence of Ni and O in the synthesized NiO NPs calcinated at (a) 300°C and (b) 400°C. Also it is observed that the NiO NPs calcinated at 300°C sample has nearly stoichiometric combination of elements in it.

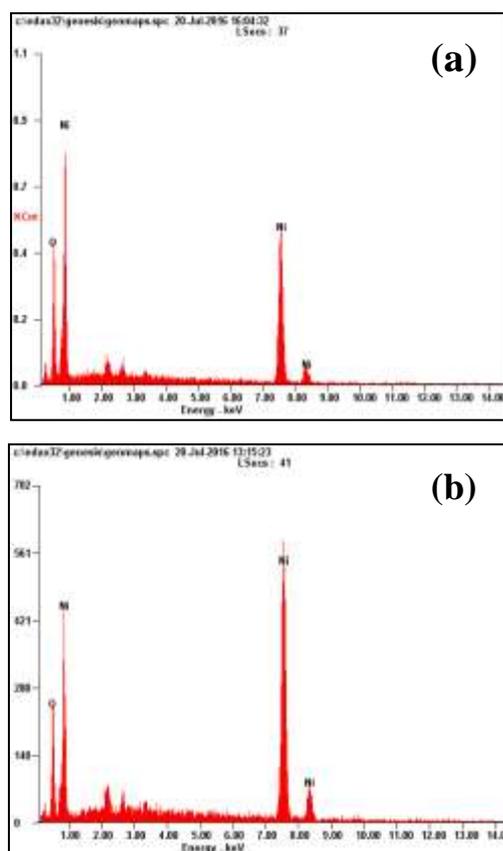


Fig. 4 EDS spectra of the NiO samples calcinated at (a) 300° C and (b) 400° C

### 3.5 PL analysis

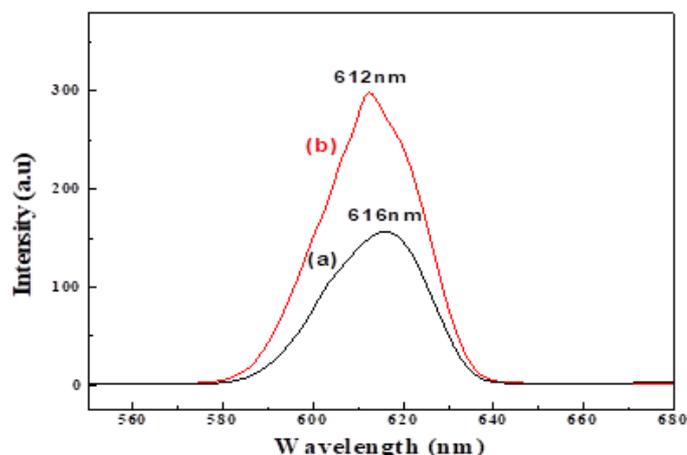


Fig. 5 PL spectra of the NiO NPs calcinated at (a) 300° C and (b) 400° C

Photoluminescence (PL) spectra of the NiO NPs calcinated at (a) 300°C and (b) 400°C are shown in figure

5. The emission peak occurred at 612 nm for the NiO NPs calcinated at 300°C and 616 nm for the NiO NPs calcinated at 400°C with an excitation wavelength 300 nm. The emission peaks of the both NiO NPs correspond to the red light emission. The intensity of the PL emission peak increases as the calcination increases. These results are in agreement with the earlier reports [19-22].

### Conclusion

The NiO NPs are successfully synthesized by chemical precipitation method at calcination temperatures 300°C and 400°C. The powder XRD studies confirmed the synthesis of NiO and revealed that both the NiO NPs, calcinated at 300°C and 400°C, have the face centered cubic structure with the average crystallite size 12.65 nm and 17.54 nm respectively. The crystallite size of NiO NPs increase with increase in the calcination temperature. The peaks at 575  $\text{cm}^{-1}$  and 424  $\text{cm}^{-1}$  in FTIR spectra of both the samples are due to the Ni-O stretching which confirms the presence of Ni and O in the prepared NiO NPs. The SEM analysis revealed that the synthesized NiO NPs are spherical in shape. The average particle sizes are found to be 24.84 nm and 27.65 nm for the NiO NPs calcinated at 300°C and 400°C respectively. The average particle sizes increase with increase in the calcination temperature. The EDS analysis confirmed the presence of Ni and O in the synthesized NiO NPs calcinated at 300°C and 400°C. The PL analysis showed that the NiO NPs, calcinated at 300°C and at 400°C, exhibited emission peaks at 616 nm and 612nm respectively. The intensity of the emission peaks increase with increasing calcination temperature.

### References

- [1] A. K. Bandyopathyay, Nanomaterials, New age international publications, ISBN: 81-224-2321-3. (2008) 1-2.
- [2] Guozhong cao, Nanostructure and nanomaterials, World scientific publications, ISBN: 86094-415-9. (2004) 1-3.
- [3] S. Shanmugam, Nanotechnology, MJP publications, ISBN: 978-81-8094-064-4. (2010) 5-7.
- [4] B. Ram Gupta, B. Uday Kompella, Nanoparticle Technology for drug delivery: Drugs and pharmaceutical sciences, ISBN: 978-1-57444-857-3. (2006) 2-9.
- [5] Bei Cheng, Yao Le, Weiquan Cai, Jiaguo Yu, J Hazard mater., 185(2011) 889-897.
- [6] Xiong Li-Zhi, He Ze-Qiang, Sun Xin-yang, J. Nat. Sci., 30 (2009) 81-85.
- [7] A. Rahdar, M. Aliahmad, Y. Azizi, J Nanostruct, 5(2015) 145-151.
- [8] M. A. Shah, Tokeer Ahmad, Principle of Nanoscience and Nanotechnology, Narosa house publication, ISBN: 978-81-8487-072-5. (2010) 48-49.
- [9] Quansheng Song Zhiyuan Tang, Hetong Guo, S.L.I Chan, J Power Sources, 112 (2002) 428-434.

- [10] Gen-Tao Zhou, Qi-zhi Yao, Xinchun Wang, C. Jimmy Yu, *Mater Chem phys.*, 98 (2006) 267-272.
- [11] G. Bharathy, P. Raji, *J Sci. Tech.*, 2 (2014).
- [12] M. Poonam, K. Wadhywani Vikram, K Panchal Nisha, *Appl. Surf. sci.*, (2015) 1- 46.
- [13] Ravi Kant sharma, Ranjana Ghose, *Superlattices Microstruct.*, 80 (2015) 169-180.
- [14] Mohammad Reza Kalaie, Amir Ali Youzbashi, Mohammad Ali Meshkot, Farzad Hosseini-Nasab, *Appl. Nanosci.*, 6 (2016) 789-795.
- [15] K. Karthick, G. Kalai selvan, M. Kanagaraj, S. Arumugam, N. Victor Jaya, *Alloys and comp.*, 509 (2011) 181-184.
- [16] M. M. Kashani Motlagh, A. A. Youzbashi, L. Sabaghzadeh, *IJPS*, 6 (2011) 1471-1476.
- [17] M. El-Kemary, N. Nagy, *Mater. Sci. Semicond. Process.*, 16(2013) 1747-1752.
- [18] K. Anandan, V.Rajendran, *Int. J. Nanosci. Nanotechnol.*, 2 (2014) 24-29.
- [19] A. Joseph sayaga Kennedy, I. Johnson, *Int. J. Chemtech Res.*, 8 (2015) 316-321.
- [20] M. Hemalatha, N. Suryanarayanan, *J. Ovonic Res.*, 8 (2012) 47-51.
- [21] B. R. Reddy, G.S Harish, C. S. Reddy, P. S. Reddy, *IJMER*, 4 (2014) 62-66.
- [22] P. A. Sheena, K.P. Priyaanka, N. A sabu, Boby sabu, T Varghese, *Nanosystems: Phys. Chem. Math.*, 5 (2014) 441-449.