Structural characterizations of Hydrothermally Synthesized NiO nanoparticles

[1*]Aparna A. Kulkarni, [2]Gotan H. Jain, [3]Rajendra P. Patil, [4]Madhavrao K. Deore,

[1,2,4]Department of Physics and Research Centre, MVP's, K.R.T. Arts, B.H. Commerce, A.M. Science College, Nashik, Shivajinagar, Gangapur Road, Nashik- 422002, India
[2]Department of Physics, MVP's, Arts, Commerce and Science College, Nandgaon, Tal. - Nandgaon, Dist. - Nashik, Maharashtra- 423106, India
[3]Bhonsala Military College, Model Colony, Nashik, Maharashtra 422005, India
Corresponding author: aparnadani17@gmail.com

Abstract

The present work is focused on the synthesis of nickel oxide (NiO) nanoparticles using hydrothermal method. The structural properties of synthesized NiO were studied by using Scanning Electron Microscopy (SEM), Energy-dispersive X-ray spectroscopy (EDAX) and X-ray diffractometer (XRD) characterizations. The SEM results shows the spherical type of surface morphology. By using BET method, the specific surface area was calculated and it was found to be 2.516 m^2/g. EDAX spectra shows the film has oxygen excess and also shows that the presence of Ni and O elements with non-stoichiometry nature. XRD results reveal that crystalline nature of particles. The obtained peaks of NiO match with JCPDS card No. 47-1049 and reveal Bunsenite face centred cubic (FCC) crystalline structure of synthesized particle. Crystallite size was estimated using Debye Scherer's formula and it is found that 29.35 nm.

Keywords: Crystalline nature, hydrothermal, nickel oxide, non-stoichiometry, and surface area.

1. Introduction

Nanoparticles are becoming increasingly important in the 21st century due to their unique properties and wide range of applications across various fields. Nanoparticles are particles that have at least one dimension (diameter, length, or width) that is on the nanometer scale, typically ranging from 1 to 100 nanometers [1, 2]. They can be composed of various materials, including metals, metal oxides, polymers, and carbon-based materials, among others. Nanoparticles exhibit unique physical, chemical, and biological properties compared to their bulk counterparts due to their small size and high surface-to-volume ratio [3, 4]. These unique properties make nanoparticles attractive for a wide range of applications in various fields, including medicine, electronics, environmental remediation, and catalysis, among others. Nanoparticles can be synthesized using various methods, such as chemical synthesis, physical methods, and biological methods, and can be engineered to have specific properties tailored for particular applications [5, 6]. Nanoparticles enable the development of advanced materials with tailored properties, such as enhanced strength, conductivity, and catalytic activity, which are crucial for technological advancements [6, 7]. Metal oxide semiconductor (MOS) nanoparticles play a crucial role in various technological advancements and are increasingly important in the current century due to their unique properties and versatile applications. MOS

are extensively used in gas sensors for detecting a wide range of gases, including toxic gases and volatile organic compounds [7-9]. Their high surface area, sensitivity, and selectivity make them ideal for environmental monitoring, industrial safety, and healthcare applications. MOS nanoparticles are used in energy storage devices, such as lithium-ion batteries and supercapacitors, due to their high specific surface area, electrical conductivity, and electrochemical stability [3, 7, 9]. They are also employed in photovoltaic devices, like solar cells, for efficient energy conversion. MOS nanoparticles are also used in optoelectronic devices, such as light-emitting diodes (LEDs), photodetectors, and transparent conductive films, due to their optical transparency, electrical conductivity, and tunable bandgap properties. MOS nanoparticles encompass a wide range of materials, each with its unique properties and potential applications [10, 11]. MOS divided into two type n and p type. The most common metal oxide semiconductor materials that found application as sensors are SnO_2, ZnO, NiO, CeO_2, Fe_2O_3, TiO_2, In_2O_3, Co_3O_4, and CuO [12-14].

Nickel oxide nanoparticles (NiO NPs) have gained significant attention in recent years due to their unique properties and potential applications in various fields [15, 16]. One of the key properties of NiO nanoparticles is their high surface area to volume ratio, which makes them ideal for use in catalysis, gas sensing, and energy storage devices. Continued research into their synthesis, properties, and applications could lead to further advancements and new opportunities in various fields [17, 18]. There are several synthesis methods for synthesis of NiO nanoparticles, including chemical precipitation, sol-gel method, and hydrothermal synthesis. Each method offers specific advantages in terms of particle size control, purity, and scalability, making it possible to tailor the nanoparticles for different applications. NiO nanoparticles have shown promise in fuel cells, supercapacitors, and environmental remediation due to their excellent electrical conductivity and catalytic activity [19-20]. Additionally, they have been explored for use in sensors for detecting toxic gases and biomolecules, as well as in biomedical applications such as drug delivery and imaging agents. The future perspectives of NiO nanoparticles are bright, with ongoing research focused on enhancing their performance and expanding their applications. Advances in the synthesis methods and surface engineering are expected to further improve the properties of NiO nanoparticles, opening up new possibilities in fields such as electronics, photonics, and water treatment. The unique properties, versatile synthesis methods, and diverse applications of NiO nanoparticles make them a promising candidate for addressing various technological and environmental challenges in the years to come.

In the present research work, the authors have focus on the synthesis and study of structural characterizations of NiO nanoparticles by hydrothermal method.

2. Synthesis of NiO nanoparticles

The synthesis of NiO particles was synthesized by using Ni $(NO_3)_2$ 6 H_2O as source of NiO. By using hydrothermal technique the NiO particles was successfully synthesized. In this method, Ni $(NO_3)_2$ 6 H_2O was dissolved in the 40 ml double distilled water. The prepared solution continuously stirred for 2 hours using magnetic stirrer. After two hours of stirring, the precipate was obtained. After data the obtained precipate was keep in Teflon lined autoclave and heated to 180 °C for 24 hrs. Then dried at 100°C overnight and then finally calcinated at 400°C for 2 hrs. After that obtained product was granded using molten and pestle for 2-3 hrs. Then final product of NiO powder was obtained.

3. Result and discussion

3.1 Scanning Electron Microscopy (SEM)

SEM characterization was carried out using Scanning Electron Microscope instrument [Model JOEL 6300 LA GERMANY]. Fig. 1 shows the SEM image of NiO nanoparticles.

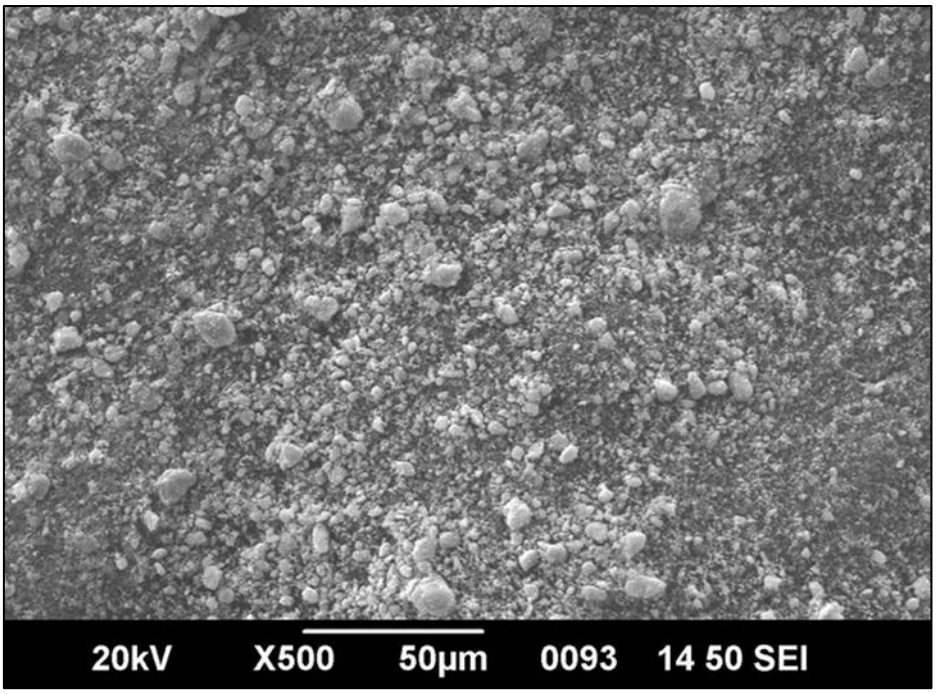

Figure 1: SEM image of NiO nanoparticles

The SEM results of NiO nanoparticles exhibit an irregular spherical shape with an average particle size of approximately 42.35 nm. SEM images also show the surface structure like a surface roughness or the presence of pores [20, 21]. The specific surface area is calculated using spherical particle diameter. The Brunauer-Emmett-Teller (BET) method [Eq.1] is used to determine the specific surface area spherical particles [22, 23]. The specific surface area for NiO nanoparticles found to be 2.516 m^2/g.

$$S_w = \frac{6}{\rho d} \quad (1)$$

Where,

S*w* - Specific surface area, *d* - Diameter of the spherical particles, and ρ – Composite density.

3.2 Energy-dispersive X-ray spectroscopy (EDAX)

Energy-dispersive X-ray spectroscopy analysis of pure NiO provides valuable information about its elemental composition, purity, and spatial distribution of elements, aiding in the characterization and quality control of the material for various applications [23]. The EDS results confirm the presence of nickel (Ni) and oxygen (O) in the synthesised powder as illustrate in Table 1.

Table 1: Elemental composition of NiO nanoparticles

Element	Weight %	Atomic %
Ni	59.63	75.84
O	40.37	24.16

3.3 X-Ray Diffraction (XRD)

XRD is a powerful technique for characterizing its crystal structure, phase purity, and structural properties, providing essential information for materials scientists and researchers studying its properties and applications [24]. The XRD characterization was carried out using an X-ray diffractometer Rigaku diffractometer [DMAX-500]. XRD data is used to calculate crystallographic parameters such as lattice constants, unit cell volume, and crystallite size, providing insights into the structural properties of material [24, 25]. The 2θ values obtained peaks were match to data files from the Joint Committee on Powder

Diffraction Standards (JCPDS). Origin 9.5 software was used to calculate the full width of half maxima (FWHM). The crystal structure for NiO was found to be face centred cubic [JCPDS card No. 47-1049] (26, 27). The major peaks were detected at 2θ = 37.28°, 43.38°, 63.12°, 75.19°, and 79.16° corresponding to the Miller indices (111), (200), (022), (311) and (222) respectively (26, 27). The prominent peak was recorded at 43.38° and corresponds (200) plane. Fig. 2 shows the XRD plot of NiO nanoparticles.

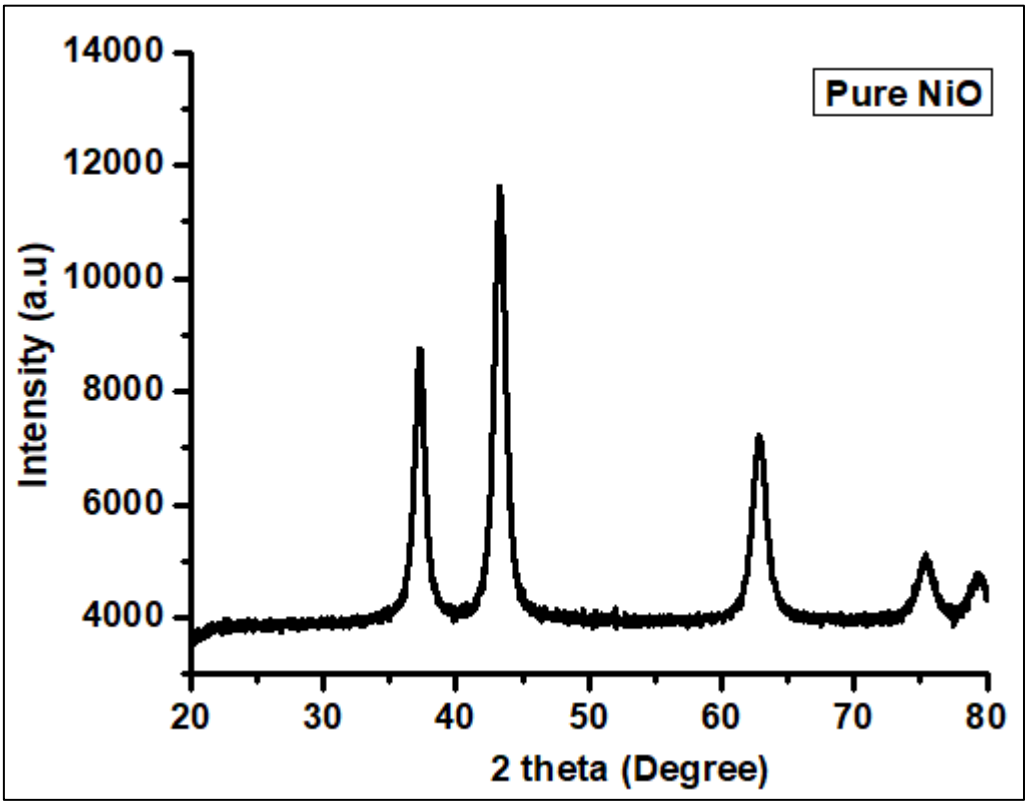

Figure 2: XRD plot of NiO nanoparticles

Debye Scherer formula, Eq. 2, is used to estimate the crystallite size [22, 23]. The crystallite size was found to be 29.35 nm (23, 28).

$$D = \frac{K\lambda}{\beta Cos\theta} \qquad (2)$$

Where,
D= Crystallite size, K= Scherrer constant (0.9), β = Full width of half maxima (FWHM), and
λ =wavelength of X source.

Conclusions

1. NiO nanoparticles successfully synthesis by eco-friendly hydrothermal technique.
2. Structural characterizations were carried out and studied using SEM, EDX and XRD.
3. XRD result shows formation of crystalline nanoparticles of NiO.
4. SEM results reveal the smooth and spherical particles formation.
5. Specific surface area was found to be 2.516 m^2/g.
6. EDX shows non-stoichiometry nature of particles.

Acknowledgment

The authors are thanks to the Research Centre in Physics, Department of Physics, MVPs KTHM College, Nashik, district- Nashik, India for providing laboratory facilities for synthesis of NiO nanoparticles. The author also thanks the Department of Physics at Savitribai Phule Pune University, Pune for providing the facilities for SEM, EDX, and XRD characterizations for this research work.

References

1. Hodes, G., 2007. When small is different: some recent advances in concepts and applications of nanoscale phenomena. *Advanced Materials*, *19*(5), pp.639-655.
2. Maurice, P.A. and Hochella, M.F., 2008. Nanoscale particles and processes: a new dimension in soil science. *Advances in agronomy*, *100*, pp.123-153.
3. Requejo-Isidro, J., Del Coso, R., Solis, J., Gonzalo, J. and Afonso, C.N., 2005. Role of surface-to-volume ratio of metal nanoparticles in optical properties of Cu: Al2O3 nanocomposite films. *Applied physics letters*, *86*(19).
4. Kwon, H.J., Shin, K., Soh, M., Chang, H., Kim, J., Lee, J., Ko, G., Kim, B.H., Kim, D. and Hyeon, T., 2018. Large-scale synthesis and medical applications of uniform-sized metal oxide nanoparticles. *Advanced Materials*, *30*(42), p.1704290.
5. Chavali, M.S. and Nikolova, M.P., 2019. Metal oxide nanoparticles and their applications in nanotechnology. *SN applied sciences*, *1*(6), p.607.
6. Naseem, T. and Durrani, T., 2021. The role of some important metal oxide nanoparticles for wastewater and antibacterial applications: A review. *Environmental Chemistry and Ecotoxicology*, *3*, pp.59-75.
7. Abo-zeid, Y. and Williams, G.R., 2020. The potential anti-infective applications of metal oxide nanoparticles: A systematic review. *Wiley Interdisciplinary Reviews: Nanomedicine and Nanobiotechnology*, *12*(2), p.e1592.
8. Oskam, G., 2006. Metal oxide nanoparticles: synthesis, characterization and application. *Journal of sol-gel science and technology*, *37*, pp.161-164.
9. Chaudhary, R.G., Bhusari, G.S., Tiple, A.D., Rai, A.R., Somkuvar, S.R., Potbhare, A.K., Lambat, T.L., Ingle, P.P. and Abdala, A.A., 2019. Metal/metal oxide nanoparticles: toxicity, applications, and future prospects. *Current Pharmaceutical Design*, *25*(37), pp.4013-4029.
10. Dizaj, S.M., Lotfipour, F., Barzegar-Jalali, M., Zarrintan, M.H. and Adibkia, K., 2014. Antimicrobial activity of the metals and metal oxide nanoparticles. *Materials Science and Engineering: C*, *44*, pp.278-284.
11. Nikolova, M.P. and Chavali, M.S., 2020. Metal oxide nanoparticles as biomedical materials. *Biomimetics*, *5*(2), p.27.
12. Sah, C.T., 1964. Characteristics of the metal-oxide-semiconductor transistors. *IEEE Transactions on Electron Devices*, *11*(7), pp.324-345.
13. Dey, A., 2018. Semiconductor metal oxide gas sensors: A review. *Materials science and Engineering: B*, *229*, pp.206-217.
14. He, H., 2020. Metal oxide semiconductors and conductors. In *Solution Processed Metal Oxide Thin Films for Electronic Applications* (pp. 7-30). Elsevier.
15. Govardhan, K. and Grace, A.N., 2016. Metal/metal oxide doped semiconductor based metal oxide gas sensors—A review. *Sensor letters*, *14*(8), pp.741-750.
16. Imran Din, M. and Rani, A., 2016. Recent advances in the synthesis and stabilization of nickel and nickel oxide nanoparticles: a green adeptness. *International journal of analytical chemistry*, *2016*.

17. Ahmad, W., Bhatt, S.C., Verma, M., Kumar, V. and Kim, H., 2022. A review on current trends in the green synthesis of nickel oxide nanoparticles, characterizations, and their applications. *Environmental Nanotechnology, Monitoring & Management*, *18*, p.100674.
18. Mokoena, T.P., Swart, H.C. and Motaung, D.E., 2019. A review on recent progress of p-type nickel oxide based gas sensors: Future perspectives. *Journal of Alloys and Compounds*, *805*, pp.267-294.
19. Ahmad, R., Bedük, T., Majhi, S.M. and Salama, K.N., 2019. One-step synthesis and decoration of nickel oxide nanosheets with gold nanoparticles by reduction method for hydrazine sensing application. *Sensors and Actuators B: Chemical*, *286*, pp.139-147.
20. Roychoudhury, A., Prateek, A., Basu, S. and Jha, S.K., 2018. Preparation and characterization of reduced graphene oxide supported nickel oxide nanoparticle-based platform for sensor applications. *Journal of Nanoparticle Research*, *20*, pp.1-17.
21. Theivasanthi, T. and Alagar, M., 2012. Chemical capping synthesis of nickel oxide nanoparticles and their characterizations studies. *arXiv preprint arXiv:1212.4595*.
22. Sinha, P., Datar, A., Jeong, C., Deng, X., Chung, Y.G. and Lin, L.C., 2019. Surface area determination of porous materials using the Brunauer–Emmett–Teller (BET) method: limitations and improvements. *The Journal of Physical Chemistry C*, *123*(33), pp.20195-20209.
23. Tupe, U.J., Zambare, M.S., Patil, A.V. and Koli, P.B., 2020. The binary oxide NiO-CuO nanocomposite based thick film sensor for the acute detection of Hydrogen Sulphide gas vapours. *Material Science Research India*, *17*(3), pp.260-269.
24. Wagh, S.L., Tupe, U.J., Patil, A.B. and Patil, A.V., 2022. Influence of Annealing Temperature on Structural and Electrical Properties of Screen Printed Lanthanum Oxide Thick Films. *Iranian Journal of Materials Science & Engineering*, *19*(4).
25. Kim, K.H., Yoshihara, Y., Abe, Y., Kawamura, M. and Kiba, T., 2017. Morphological characterization of sphere-like structured ZnO-NiO nanocomposites with annealing temperatures. *Materials Letters*, *186*, pp.364-367.
26. Dang, T.T.L., Tonezzer, M. and Nguyen, V.H., 2015. Hydrothermal growth and hydrogen selective sensing of nickel oxide nanowires. *Journal of nanomaterials*, *2015*, pp.6-6.
27. Zhang, G., Yu, L., Hoster, H.E. and Lou, X.W.D., 2013. Synthesis of one-dimensional hierarchical NiO hollow nanostructures with enhanced supercapacitive performance. *Nanoscale*, *5*(3), pp.877-881.
28. Kim, K.H., Mikami, M., Abe, Y., Kawamura, M. and Kiba, T., 2018. Morphological evolution of self-supporting nickel hydroxide nanostructures prepared by a facile wet-chemical method. *Thin Solid Films*, *654*, pp.49-53.