Research Article

Journal of Environmental Nanotechnology

Impact of Reducing Agents on the Synthesis of Nickel Oxide Nanoparticles by Chemical Precipitation Method

V. Kayathri, K. Kousalya, A. Mafeena, M. Monisha, S. Naga Nandhini, J. Balavijayalakshmi*
Department of Physics, PSGR Krishnammal College for Women, Coimbatore, TN, India
Received: 13.01.2021 Accepted: 03.03.2021 Published: 30.03.2021
*balavijayalakshmiroopa@gmail.com

ABSTRACT

This present work reports the synthesis ad characterization of Nickel oxide nanoparticles by chemical precipitation method from nickel chloride by using two different reducing agents, viz. ammonia (NH_3) and sodium hydroxide (NaOH). The prepared nanoparticles were characterized by X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Field Emission-Scanning Electron Microscopy (FE-SEM) and Energy Dispersive X-ray analysis (EDAX) techniques. XRD analysis showed that the prepared nanoparticles were crystalline in nature and the average crystallite size for NiO nanoparticles with NaOH and NH_3 were found to be 25.3 nm and 27.54 nm, respectively. FE-SEM analysis revealed that the prepared nanoparticles were spherical in shape with agglomeration. The presence of functional groups of the prepared nanoparticles was confirmed by FT-IR analysis. The band at 450 cm^{-1} corresponds to the stretching vibration on Ni-OH, confirming the presence of NiO and the elemental analysis confirmed the presence of nickel and oxygen elements in the prepared nickel oxide nanoparticles without any impurities. The synthesized nickel oxide nanoparticles may be used in micro-supercapacitors, electrochromic coatings and chemical sensing devices.

Keywords: Chemical precipitation; Nickel oxide; Reducing agent.

1. INTRODUCTION

Nickel oxide (NiO) is the most widely investigated transition metal oxide. It is a NaCl-type antiferromagnetic oxide semiconductor. It offers promising candidature for many applications such as solar thermal absorber, the catalyst for O_2 evolution, photo-electrolysis and electrochromic device. Nickel oxide is also a well-studied material as a positive electrode in batteries (Ibraheem *et al.* 2019; Lingaraju *et al.* 2019). Pure stoichiometric NiO crystals are perfect insulators. Several efforts have been made to explain the insulating behavior of NiO. Appreciable conductivity can be achieved in NiO by creating Ni vacancies or substituting Li for Ni at Ni sites. The most attractive features of NiO are excellent durability, electrochemical stability and low material cost. It is a promising ion storage material in terms of cyclic stability, large span optical density and the possibility of manufacturing by a variety of techniques. Nickel oxide has a good electrochemical property, low toxicity and low cost, making them suitable for many applications (Patil *et al.* 2002; Nassar *et al.* 2017). In this present work, nickel oxide nanoparticles were synthesized by chemical precipitation method from Nickel chloride by using two different reducing agents such as ammonia (NH_3) and sodium hydroxide (NaOH).

The chosen metal oxide nanoparticle is an important transition metal oxide with a cubic lattice structure and of p-type semiconducting material with a bandgap of 4.0 eV. The purpose of the study is to explore the feasibility to synthesize NiO nanoparticles by chemical precipitation method from Nickel chloride by using two different reducing agents such as ammonia (NH_3) and sodium hydroxide (NaOH). The results obtained by characterization techniques that involve X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Field Emission-Scanning Electron Microscopy (FE-SEM) and Energy Dispersive X-ray Analysis (EDAX) were reported.

2. MATERIAL AND EXPERIMENTS

2.1 Materials

Nickel chloride, sodium hydroxide (NaOH) and Ammonia (NH_3) were purchased from Sigma Aldrich and used without further purification.

2.2 Synthesis of Nickel Oxide Nanoparticles

NiO nanoparticles were prepared by the chemical precipitation method. 8.5 g of nickel chloride was dissolved in 80 ml of distilled water. A light green

color solution was obtained, which is sonicated for an hour and then the particles were agitated using ultrasonic vibrations (Singh *et al.* 2016). The sonicated solution was stirred into which NaOH is added drop by drop as a reducing agent to maintain a pH value of about 8. The process of stirring was carried with 450 rpm at 45 °C for about 4 hours. Then the solution was kept undisturbed for 24 hours to obtain a green precipitate which settled in the bottom of the beaker. The filtrate was centrifuged several times at 2000 rpm with each cycle of duration 10 minutes using centrifugal force to separate fluid of different densities. After the process of centrifuge, the resulting white precipitate was cleaned and dried at 60 °C for about 4 hours to allow ionic movements by heating the particles at elevated temperature. The precipitate was calcinated under 450 °C for about 4 hours. The obtained particles were grained into fine particles of powder form using mortar and pestle. The same procedure was repeated to synthesize Nickel Oxide nanoparticles using ammonia as a reducing agent (T. Sone *et al.* 2016; Shanmugapriya *et al.* 2019).

2.3 Characterization Techniques

The prepared NiO nanoparticles' crystalline structure was examined using an XPERT-3 PANalytical Diffractometer. Shimadzu IR affinity-1 was used to record FT-IR spectra for the presence of nanoparticles. The morphology and microstructure of the sample were studied using Field Emission Scanning Electron Microscopy (FESEM), with the help of ZEISS-SIGMA. The elemental composition was tested using Energy Dispersive X-ray Spectroscopy (EDX).

3. RESULTS AND DISCUSSIONS

3.1 XRD Analysis

The X-ray diffraction patterns of prepared NiO nanoparticles using ammonia (NH_3) and sodium hydroxide (NaOH) as reducing agents is shown in Fig. 1 (a-b). Fig. 1(a) shows the diffraction peaks at 2θ values of 37.38°, 43.42°, 63.02°and 75.53°, corresponding to (111), (200), (220) and (311) planes respectively and Fig. 1(b) shows the diffraction peaks at 2θ values of 38.2°, 43.4°, 63.0°and 75.3° corresponding to (111), (200), (220) and (311) planes respectively and are well-matched with JCPDS card number 47-104, confirming the presence of NiO nanoparticles. The crystallite sizes for prepared nanoparticles of NiO(NH_3) and NiO(NaOH) are found to be about 27.54 nm and 25.3 nm, respectively, by using the Scherrer formula (Shanmugapriya *et al.* 2019).

3.2 FT-IR Spectral Analysis

The FT-IR spectrum of the prepared NiO nanoparticles with ammonia and sodium hydroxide as reducing agents is shown in Fig. 2(a-b). It is observed that the broad absorption bands at 3440 cm^{-1} and 3460 cm^{-1} can be attributed to the O-H stretching vibrations of NiO (NH_3) and NiO (NaOH), respectively. The bands at 1647 cm^{-1} and 1630 cm^{-1} corresponds to the H-O-H stretching vibration of NiO (NH_3) and NiO (NaOH), respectively (Bhavani *et al.* 2017). The bands at 683 cm^{-1} and 702 cm^{-1} represent the Ni-OH band stretching vibrations, where the oxygen combined with nickel-metal as a binary chemical compound of NiO (NH_3) and NiO (NaOH), respectively. The bands at 428 cm^{-1} and 436 cm^{-1} represent the Ni-O band stretching vibrations of NiO(NH_3) and NiO(NaOH), respectively.

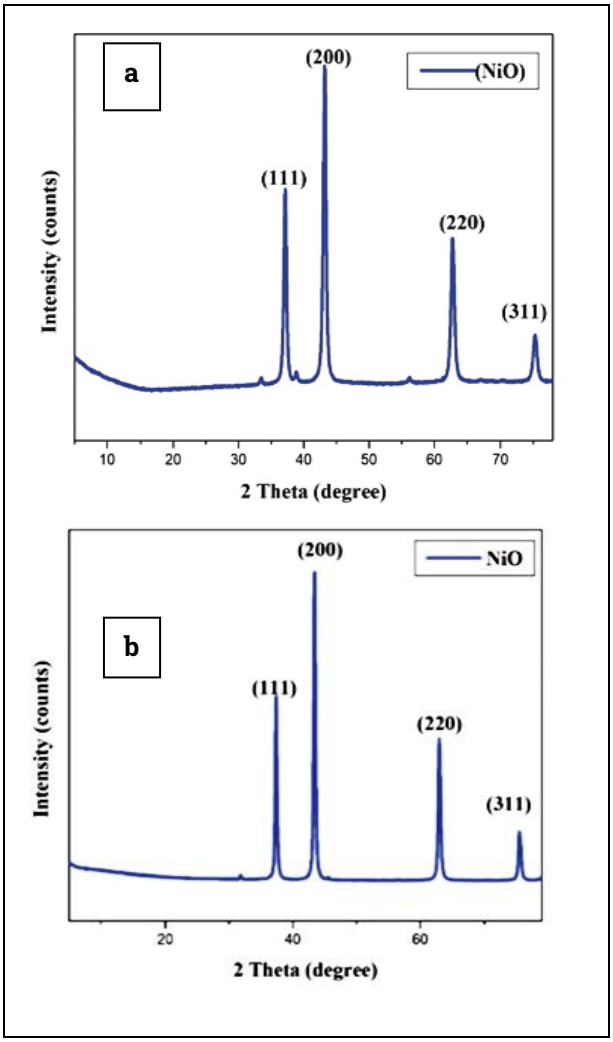


Fig. 1: XRD spectra of NiO nanoparticles using (a) Ammonia and (b) Sodium hydroxide as reducing agents

Thus, the presence of NiO functional groups can be confirmed using FT-IR spectral analysis (Ali *et al.* 2018).

Fig. 3(a-b) shows the Field-Emission Scanning Electron Microscope images of NiO nanoparticles using NH_3 as reducing agent and Fig. 3 (c-d) shows FE-SEM images of NiO nanoparticles using NaOH as reducing agent. Fig. 3 (a-d) shows the morphological analysis of the prepared NiO nanoparticles, which were spherical in shape with agglomeration (Sun *et al.* 2017; Lassoued *et al.* 2018).

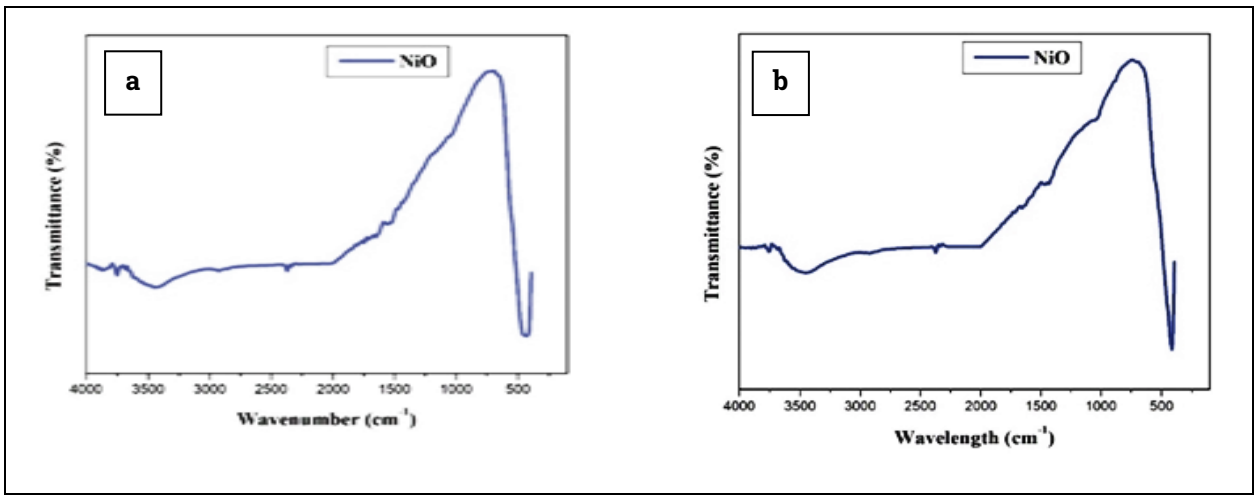


Fig. 2: FT-IR spectra of NiO nanoparticles using (a) Ammonia and (b) Sodium hydroxide as reducing agents

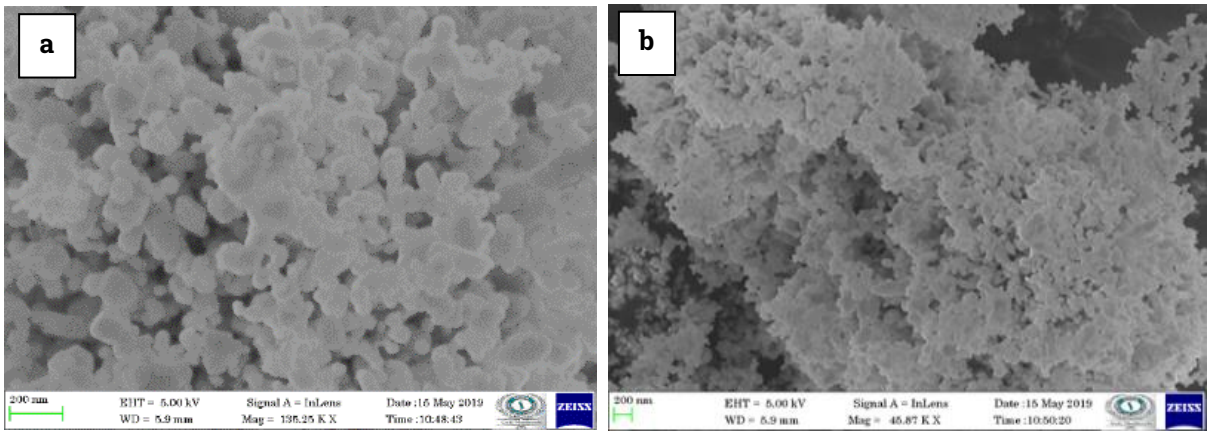


Fig. 3 (a-b): FE-SEM images of NiO nanoparticles using ammonia as reducing agent

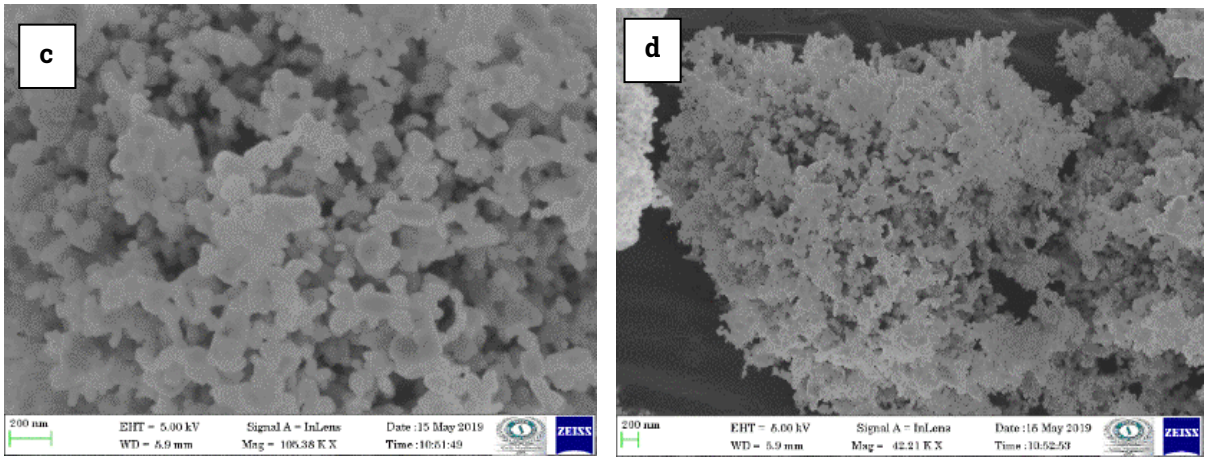


Fig. 3 (c-d): FE-SEM images of NiO nanoparticles using sodium hydroxide as reducing agent

3.4 EDAX Analysis

Fig. 4 (a-b) shows the Energy Dispersive X-ray spectral analysis of NiO nanoparticles using NH_3 and NaOH as reducing agents, respectively, performed to know the elemental presence of NiO nanoparticles (Taghizadeh *et al.* 2016; Gandhi *et al.* 2016). Fig. 4 (a) shows that the element O K has an atomic weight of 52.15 % and Ni K has an atomic weight of 47.85 %, whereas Fig. 4 (b) shows that the element O K has an atomic weight of 76.73 % and Ni K has the atomic weight of 23.27 % and thereby confirms the elemental presence of nickel oxide nanoparticles without any impurities (Kavitha *et al.* 2016).

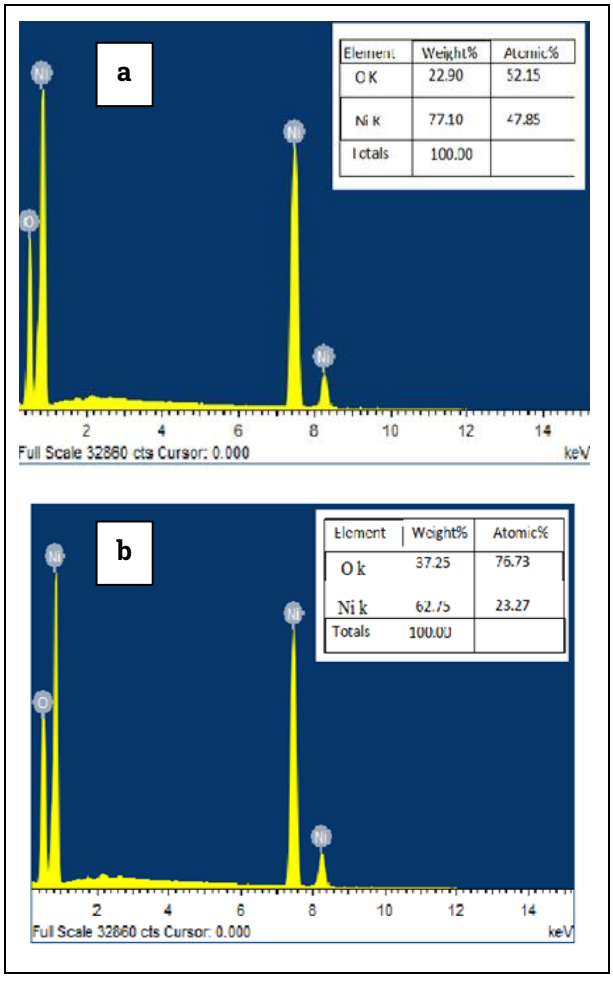


Fig. 4: EDAX analysis for NiO nanoparticles using (a) Ammonia and (b) Sodium hydroxide as reducing agents

4. CONCLUSION

Nickel Oxide nanoparticles have been successfully synthesized by chemical precipitation method from Nickel chloride by using two different reducing agents such as ammonia (NH_3) and sodium hydroxide (NaOH). The prepared nanoparticles were characterized by X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Field Emission-Scanning Electron Microscopy (FE-SEM) and Energy Dispersive X-ray Analysis (EDAX) techniques. XRD analysis showed that the prepared nanoparticles were crystalline in nature and the average crystallite size for NiO nanoparticles with NaOH and NH_3 as reducing agents were found to be 25.3 nm and 27.54 nm, respectively. FE-SEM analysis revealed that the prepared nanoparticles were spherical in shape with agglomeration. The presence of functional groups of the prepared nanoparticles was confirmed by using FT-IR analysis. The band at 450 cm^{-1} corresponds to the stretching vibration of Ni-OH, confirming the presence of NiO; the elemental analysis confirmed the presence of nickel and oxygen elements in the prepared nickel oxide nanoparticles without any impurities. It is inferred that the nickel oxide prepared using sodium hydroxide as a reducing agent is better than using ammonia, as evident from XRD analysis due to the reduction in the crystallite size and also because of its environment-friendliness and non-toxicity. The prepared Nickel oxide (NiO) can be employed in capacitor-inductor devices, tuned circuits, transparent heat mirrors, thermistor, batteries, micro-supercapacitors, electrochromic and chemical or temperature sensing devices.

FUNDING

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

COPYRIGHT

This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC-BY) license (http://creativecommons.org/licenses/by/4.0/).

REFERENCES

Ali, A. A., El Fadaly, E. and Ahmed, I. S., Near-infrared reflecting blue inorganic nano-pigment based on cobalt aluminate spinel via combustion synthesis method, *Dye. Pigment.*, 158, 451–462 (2018). https://dx.doi.org/10.1016/j.dyepig.2018.05.058

Bhavani, P., Rajababu, C. H., Arif, M. D., Reddy, I. V. S. and Reddy, N. R., Synthesis of high saturation magnetic iron oxide nanomaterials via low temperature hydrothermal method, *J. Magn. Magn. Mater.*, 426, 459–466 (2017). https://dx.doi.org/10.1016/j.jmmm.2016.09.049

Gandhi, A. C., Cheng, H.-Y., Chang, Y.-M. and Lin, J. G., Size confined magnetic phase in NiO nanoparticles, *Mater. Res. Express*, 3(3), 035017 (2016). https://dx.doi.org/10.1088/2053-1591/3/3/035017

Ibraheem, F., Aziz, M. H., Fatima, M., Shaheen, F., Ali, S. M. and Huang, Q., In vitro cytotoxicity, MMP and ROS activity of green synthesized nickel oxide nanoparticles using extract of *Terminalia chebula* against MCF-7 cells, *Mater. Lett.*, 234, 129–133 (2019). https://dx.doi.org/10.1016/j.matlet.2018.09.075

Kavitha, B., Nirmala, M. and Pavithra, A., Annealing effect on nickel oxide nanoparticles synthesized by sol-gel method, *World Sci. News*, 52, 118–129 (2016).

Lassoued, A., Lassoued, M. S., Dkhil, B., Ammar, S. and Gadri, A., Synthesis, photoluminescence and Magnetic properties of iron oxide (α-Fe_2O_3) nanoparticles through precipitation or hydrothermal methods, *Phys. E Low-dimensional Syst. Nanostruct.*, 101, 212–219 (2018). https://dx.doi.org/10.1016/j.physe.2018.04.009

Lingaraju, K., Naika, H. R., Nagabhushana, H. and Nagaraju, G., *Euphorbia heterophylla* (L.) mediated fabrication of ZnO NPs: Characterization and evaluation of antibacterial and anticancer properties, *Biocatal. Agric. Biotechnol.*, 18, 100894 (2019). https://dx.doi.org/10.1016/j.bcab.2018.10.011

Nassar, M. Y., Aly, H. M., Abdelrahman, E. A. and Moustafa, M. E., Synthesis, characterization, and biological activity of some novel Schiff bases and their Co(II) and Ni(II) complexes: A new route for Co_3O_4 and NiO nanoparticles for photocatalytic degradation of methylene blue dye, *J. Mol. Struct.*, 1143, 462–471 (2017). https://dx.doi.org/10.1016/j.molstruc.2017.04.118

Patil, P. and Kadam, L. , Preparation and characterization of spray pyrolyzed nickel oxide (NiO) thin films, *Appl. Surf. Sci.*, 199(1–4), 211–221 (2002). https://dx.doi.org/10.1016/S0169-4332(02)00839-5

Shanmugapriya, T. and Balavijayalakshmi, J., Preparation and characterization of nitrogen doped graphene oxide/nickel oxide nanocomposites for dye sensitized solar cell applications, *Adv. Appl. Res.*, 11(1), 34 (2019). https://dx.doi.org/10.5958/2349-2104.2019.00006.8

Singh, P., Kim, Y.-J., Zhang, D. and Yang, D.-C., Biological synthesis of nanoparticles from plants and microorganisms, *Trends Biotechnol.*, 34(7), 588–599 (2016). https://dx.doi.org/10.1016/j.tibtech.2016.02.006

Sun, D.-L., Zhao, B.-W., Liu, J.-B., Wang, H. and Yan, H., Application of nickel oxide nanoparticles in electrochromic materials, *Ionics (Kiel).*, 23(6), 1509–1515 (2017). https://dx.doi.org/10.1007/s11581-017-1974-4

Sone, B. T., Fuku, X. G. and Maaza, M., Physical and electrochemical properties of green synthesized bunsenite NiO nanoparticles via *Callistemon viminalis* extracts, *Int. J. Electrochem.* Sci., 11, 8204–8220(2016). https://dx.doi.org/10.20964/2016.10.17

Taghizadeh, F., The study of structural and magnetic properties of NiO nanoparticles, *Opt. Photonics J.*, 06(08), 164–169 (2016). https://dx.doi.org/10.4236/opj.2016.68B027