



# Nanostructured amorphous nickel oxide with enhanced antioxidant activity



G. Madhu<sup>a,b</sup>, V. Biju<sup>a,\*</sup>

<sup>a</sup> Department of Physics, University of Kerala, Kariavattom Campus, Thiruvananthapuram, Kerala 695581, India

<sup>b</sup> Department of Physics, University College, Thiruvananthapuram, Kerala 695034, India

## ARTICLE INFO

### Article history:

Received 25 October 2014

Received in revised form 10 February 2015

Accepted 22 February 2015

Available online 26 February 2015

### Keywords:

Amorphous materials

Chemical synthesis

X-ray diffraction

Photoelectron spectroscopy

Transmission Electron Microscopy

## ABSTRACT

Nanostructured amorphous nickel oxide was synthesized by the thermal decomposition of nickel chloride–ethanol amine complex. The X-ray diffraction and Transmission Electron Microscopic studies established the amorphous nature of the sample. The Fourier Transform Infrared, Scanning Electron Microscopy, Energy Dispersive and X-ray Photoelectron Spectroscopic studies of the sample revealed the formation of NiO. The specific surface area of the sample is measured using Brunauer–Emmett–Teller analysis and the mesoporous nature of the sample is established through Barrett–Joyner–Halenda pore size distribution analysis. The antioxidant activity of the amorphous sample measured by 1,1-diphenyl-2-picryl hydrazyl (DPPH) scavenging is found to be nearly twice greater than that reported for nanocrystalline NiO samples. The estimated radical scavenging activity of the sample is correlated with the DC conductivity values measured in vacuum and air ambience. The enhanced antioxidant activity of the amorphous NiO is accounted by the increase in the concentration of O<sup>2-</sup> vacancies and the specific surface area. The Ni 2p and O 1s X-ray Photoelectron Spectroscopic studies of the sample support the inference.

© 2015 Elsevier B.V. All rights reserved.

## 1. Introduction

Nickel oxide (NiO) with face centered cubic structure (space group Fm3m) is an antiferromagnet with high Neel temperature ( $T_N = 523$  K) [1]. At ordinary temperatures, it exhibit enhanced electrical conductivity, electrochemical property, superparamagnetism/superantiferromagnetism, etc. [2–4]. The varied physical and chemical properties of NiO are primarily determined by the localized 3d band of Ni<sup>2+</sup> ions [5]. Moreover, the presence of defects such as cation/anion vacancies and stoichiometry of the sample play an important role in determining its properties. Nanostructured NiO find applications in diverse fields such as temperature sensors, smart windows, switching devices, super-capacitors, Li-ion batteries,  $\gamma$ -sensors, and spin valves [6–8]. The biomedical applications of nanostructured NiO include contrasting agents for magnetic resonance imaging, carriers, adsorbents, etc. [9]. Ni/NiO nanoparticles stabilized with amphipathic peptide have potential properties for the treatment of Alzheimer's disease [10].

Saikia et al. had first reported the antioxidant activity of nanostructured NiO with crystallite sizes in the range 10–60 nm and they commented little on the mechanism behind the observed

antioxidant activity [11]. In our previous report, we have done a detailed investigation on the antioxidant activity of nanostructured NiO samples with different average crystallite sizes, viz., 32, 37, 40 and 45 nm, synthesized through a facile chemical route [12]. The antioxidant activity was quantified by the scavenging ability of free radical DPPH (1,1-diphenyl-2-picryl hydrazyl) being monitored by UV–visible absorption spectrum. The use of X-ray Photoelectron Spectroscopy (XPS) and temperature dependent DC electrical conductivity measurements of the samples in controlled environments–vacuum and air ambience–revealed that the antioxidant activity of nanostructured NiO is due to the presence of O<sup>2-</sup> vacancies associated with the surface of the nanoparticles. On annealing the sample in air ambience at higher temperatures, the antioxidant activity decreased markedly due to the filling up of O<sup>2-</sup> vacancies [12]. This paper reports for the first time the antioxidant activity of nanostructured amorphous NiO which is found to be much larger than that of nanocrystalline NiO.

## 2. Experimental

We had previously reported the synthesis of nanostructured NiO samples in the size range 32–45 nm using nickel chloride [NiCl<sub>2</sub>·4H<sub>2</sub>O] and ethanol amine [CH<sub>2</sub>–(OH)–CH<sub>2</sub>–NH<sub>2</sub>] [12]. In the first step, nickel chloride–ethanol amine (NiCl<sub>2</sub>–EA) complex was synthesized. The Thermo Gravimetric Analysis (TGA) of the complex revealed that the decomposition of the complex starts just above

\* Corresponding author. Tel.: +91 471 2308920.

E-mail address: [bijunano@gmail.com](mailto:bijunano@gmail.com) (V. Biju).

300 °C. Nanocrystalline NiO sample with an average crystallite size of 32 nm was synthesized by the thermal decomposition of the complex at 350 °C for 8 h (sample code-N3508) [12]. Further, nanocrystalline NiO samples with average crystallite sizes of 37 nm (sample code-N400), 40 nm (sample code-N500) and 45 nm (sample code-N600) were obtained by 2 h annealing of the as-prepared sample at 400, 500 and 600 °C respectively. In the present study, the thermal treatment of the NiCl<sub>2</sub>-EA complex was done at 350 °C, but the duration was reduced to 4 h (sample code-N3504).

The structural characterization of the sample was done using X-ray diffraction (XRD) and Transmission Electron Microscopy (TEM) techniques. The XRD pattern of the sample was recorded using an X'Pert Pro X-ray Diffractometer with Cu K $\alpha$  source ( $\lambda = 1.540560 \text{ \AA}$ ). The TEM image and Selected Area Electron Diffraction (SAED) pattern of the sample were recorded using a Philips CM-200 Electron Microscope. SHIMADZU FTIR-300 spectrometer was used to record the FTIR spectra of the samples in the range 400–4000 cm<sup>-1</sup>. The SEM images of the samples were collected using JOEL Model JSM-6390 Scanning Electron Microscope and Energy Dispersive Spectra (EDS) of the samples were collected using a JEOL Model JED-2300 spectrometer. M/s SPECS make X-ray Photoelectron spectrometer under an operating pressure of  $6 \times 10^{-8}$  mbar with Al K $\alpha$  (1486.7 eV) radiation as the source was used to record the X-ray Photoelectron Spectrum (XPS) of the samples. The

specific surface area (BET) of the samples was measured by nitrogen adsorption-desorption measurements at the liquid nitrogen temperature using a Gemini 2375 volumetric apparatus and the result was used to determine the particle size of the samples. The BJH method was used to calculate the pore size distribution of the samples using the desorption data. The antioxidant activities of the samples were estimated by the modified DPPH scavenging technique with the help of a SHIMADZU UV-2550 double beam spectrophotometer in the range 450–550 nm. The details of the measurement of antioxidant activity have already been reported [12]. The DC electrical conductivities of the samples consolidated in the form of cylindrical pellet were measured in a dielectric cell in vacuum (0.05 mbar) and air ambience at different temperatures ranging from 303 to 423 K using a KEITHLEY 2400 source meter.

### 3. Results and discussions

#### 3.1. Structural characterizations

The XRD pattern and FTIR spectra of the sample N3504 are shown in Fig. 1(A) and (B). For comparison, XRD pattern and FTIR spectra of the crystalline samples (N3508, N400, N500 and N600)

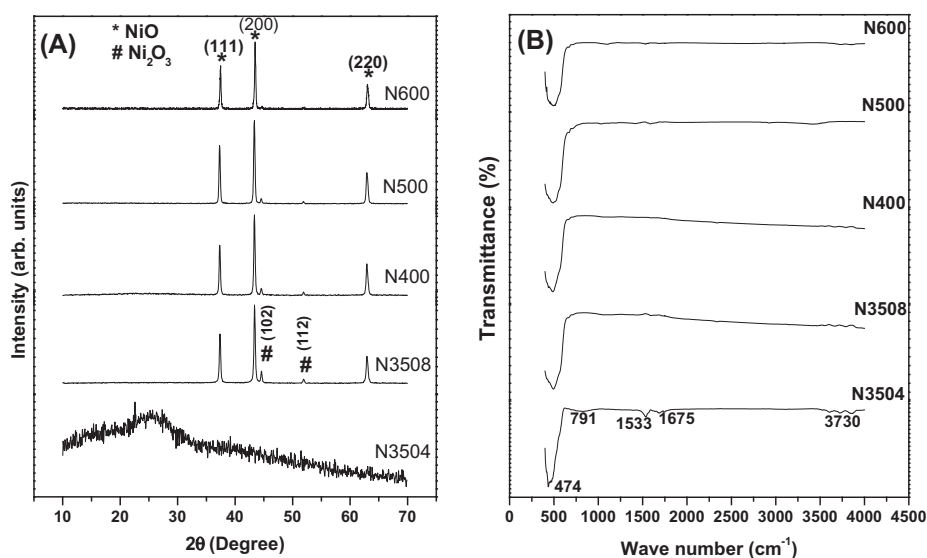


Fig. 1. X-ray diffraction pattern (A) and FTIR spectra (B) of amorphous and crystalline NiO samples.

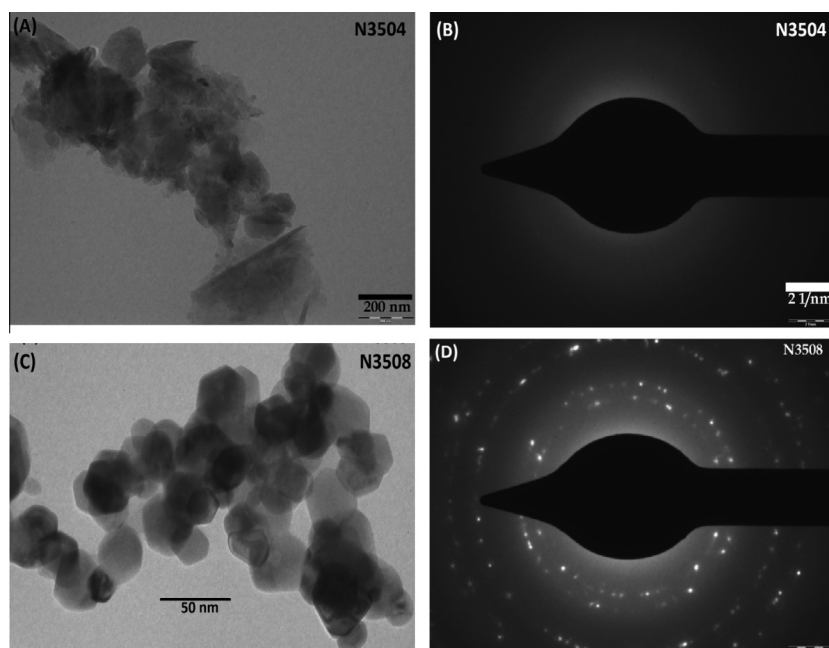


Fig. 2. TEM image and SAED pattern of samples N3504 and N3508.

Download English Version:

<https://daneshyari.com/en/article/1609464>

Download Persian Version:

<https://daneshyari.com/article/1609464>

[Daneshyari.com](https://daneshyari.com)