Journal of Advanced Chemical Sciences 2(2) (2016) 230–232

ISSN: 2394-5311

Contents List available at JACS Directory

Journal of Advanced Chemical Sciences

journal homepage: www.jacsdirectory.com/jacs

Hydrothermal Synthesis, Characterization and Antibacterial Activity of NiO Nanoparticles

A. Santhoshkumar, Helen P. Kavitha*, R. Suresh

Department of Chemistry, SRM University, Ramapuram Campus, Chennai – 600 089, Tamil Nadu, India.

ARTICLE DETAILS

Article history:
Received 10 January 2016
Accepted 22 January 2016
Available online 24 February 2016

Keywords:
NiO
Nanoplates
Triethylamine
Antibacterial Activity

ABSTRACT

NiO nanoplates were synthesized by treating nickel sulphate and triethylamine (TEA) followed by annealing at 600 °C for 6 h. The formation of cubic phase NiO and crystalline nature were confirmed by X-ray diffraction (XRD) analysis. Fourier transform infrared (FTIR) spectroscopy confirms the existence of Ni-O bond in the sample. Light harvesting behaviour and band gap of NiO nanoplates were determined by UV-Visible spectroscopy. Field emission scanning electron microscopy (FE-SEM) and high resolution transmission electron microscopy (HR-TEM) reveal that the synthesized sample has plate-like morphology. Furthermore, antibacterial activity of NiO nanoplates was investigated and maximum zone of inhibition was found in 80 mg/mL NiO nanoplates against selected organisms.

1. Introduction

Semiconductor nanomaterials have received much attention owing to their excellent magnetic, electronic, chemical, optical and thermal properties [1]. Among them, nickel oxide (NiO) is one of the important p-type semiconductor materials. It has being attracting considerable attention by the researchers due to its unique magnetic, electronic, mechanical, and optical properties [2]. It can be used as smart windows, catalyst, supercapacitor, electrode, and electrochromic devices [3-5]. Because of its potential applications, variety of methods such as thermal evaporation, sputtering, sol–gel method, and pulsed laser deposition have been reported [6-8]. However, hydrothermal method is a versatile route to prepare NiO nanomaterials because of its advantages like easy procedure, low cost, and eco-friendly method.

In this paper, we adapt wet chemical method for the synthesis of NiO nanoplates. NiO powder was characterized by XRD, FTIR, DRS UV-Vis, FE-SEM and HR-TEM. Antibacterial activity of NiO nanoparticles was tested against some selected pathogenic bacteria. Based on our obtained results, NiO nanoplates have effective antibacterial activity.

2. Experimental Methods

2.1 Materials

Nickel sulphate ($NiSO_4$), and triethylamine was purchased from Qualigens and used as received. Double distilled water was used as the solvent for the experiment.

2.2 Procedure

A wet chemical method was employed for the synthesis of NiO nanoplates. The procedure is as follows: About 2.6 g of $NiSO_4$ was dissolved in 100 mL double distilled water. To the above solution, 50 mL of 0.01 M aqueous TEA solution was slowly added with constant stirring condition. The resulting mixture was continuously stirred for 2 h and the obtained precipitate was filtered and washed with adequate amount of water. The air dried powder was calcined at 600 °C for 6 h in a muffle furnace.

2.3 Characterization Techniques

The structure, metal-oxygen stretching vibration and band gap of the synthesized sample were examined by a Rich Siefert 3000 diffractometer with Cu-Kα_1 radiation (λ = 1.5406 Å), a Schimadzu FT-IR 8300 series instrument and CARY 5E UV–Vis-NIR spectrophotometer respectively. The morphology was analyzed by using a Hitachi-SU6600 FESEM instrument and JEOL JEM 2100 high resolution transmission electron microscope.

2.4. Determination of Antibacterial Activity of NiO Nanoplates

The experiments on the antibacterial activity of NiO nanoparticles were carried out by agar well diffusion method [9]. Antibacterial activity of the synthesized NiO nanoplates was tested against *Bacillus subtilis* (*B. subtilis*), *Staphylococcus aureus* (*S. aureus*), *Escherichia coli* (*E. coli*) and *Proteus vulgaris (P. vulgaris)*. All the bacteria were grown in nutrient broth medium. A small amount of sample was gently pushed over the nutrient agar plate for intimate contact of NiO nanoplates with bacterial cell. The plates were incubated at 37 °C for 24 h.

3. Result and Discussion

3.1 Characterization of NiO Powder

Fig. 1 exhibits the X-ray diffraction pattern of the black powder after thermal decomposition of Ni-TEA complex at 600 °C for 6 h. All the diffraction peaks at 37.2°, 43.5°, 63.2°, 75.3°, 79.2° are belonged to cubic NiO (JCPDS No.: 73-1523). Significant peak broadening of NiO powder indicates that the ultra-fine nature of the synthesized powder [10]. The average crystallite size of NiO powder was calculated using Debye–Scherrer equation [11]. The estimated average crystallite size of NiO is 92 nm which confirms the formation of nanocrystalline NiO.

Fig. 2 presents the FTIR spectrum of synthesized NiO powder. In general, the FTIR bands due to metal - oxygen occurs in the region of 400-850 cm^{-1}. Fig. 2 shows peak at 455 cm^{-1} which corresponds to Ni–O stretching vibration [12]. This result is well consistent with other previously published reports. The bands at 3241 cm^{-1} and 1632 cm^{-1} are assigned O–H stretching vibration of surface adsorbed water molecule [13].

*Corresponding Author
Email Address: helenkavitha1971@gmail.com (Helen P. Kavitha)

Cite this Article as: A. Santhoshkumar, Helen P. Kavitha, R. Suresh, Hydrothermal synthesis, characterization and antibacterial activity of NiO nanoparticles, J. Adv. Chem. Sci. 2(2) (2016) 230–232.

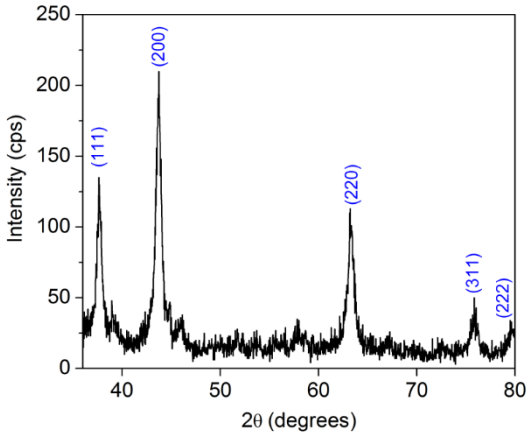

Fig. 1 XRD pattern of NiO nanoplates

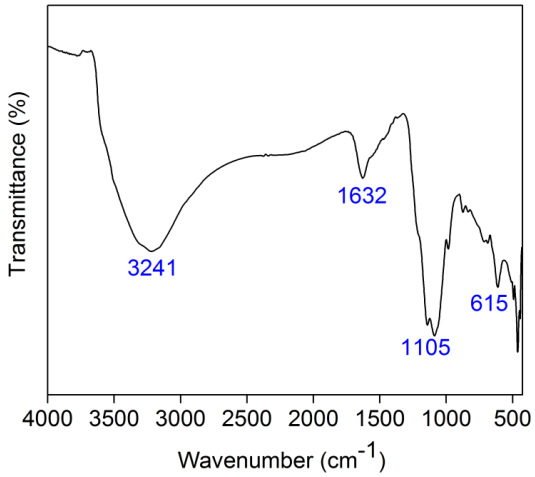

Fig. 2 FT-IR spectrum of NiO nanoplates

3.2 Morphology of NiO Powder

FE-SEM and HR-TEM techniques were used to examine the shape and size of the synthesized NiO powder. Fig. 3a-b shows FE-SEM images of NiO powder which reveals that the sample was agglomerated by nanoparticles with irregular shape. The average particles size was found to be 96 nm. The chemical composition of NiO nanoparticles was investigated by energy dispersive X-ray spectroscopy (EDS) (Fig. 3d). It shows that the NiO nanoparticles are composed of Ni and O only. The atomic ratio of Ni and O was close to 1:1 ratio which is in agreement with the stoichiometric properties of NiO.

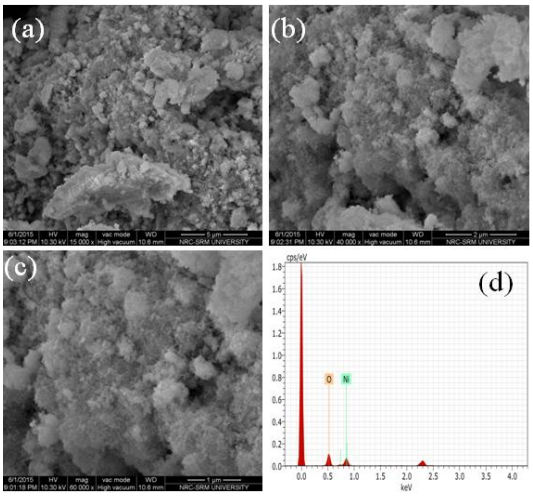

Fig. 3 (a-c) FE-SEM image of NiO nanoplates at different magnification and (d) EDS of NiO nanoparticles

In order to confirm the morphology of NiO, HR-TEM analysis was carried out. Fig. 4a-d shows the HR-TEM images of NiO nanoparticles with different magnification. HR-TEM images displays the formation of plate-like NiO nanoparticles with different size and shape. Also, particles are porous in nature which is due to liberation of gaseous product during calcination process [14]. A schematic picture of the formation of NiO nanoplates is shown in Fig. 5 and the mechanism can be explained as follows: Addition of TEA to Ni^{2+} ions solution, immediately Ni^{2+}-TEA complex nuclei will be formed which will undergo aggregation to form smaller Ni^{2+}-TEA particles. Addition of TEA is very slower. Therefore, Ni^{2+} ions may adsorb on the ledge site of Ni^{2+}-TEA particles followed by TEA adsorption and the growth of Ni-TEA particles was found to be in a linear fashion [15]. After the calcination of Ni^{2+}-TEA particles at 600 °C for 6 h, we obtained NiO nanoparticles as evidenced by XRD and FTIR results.

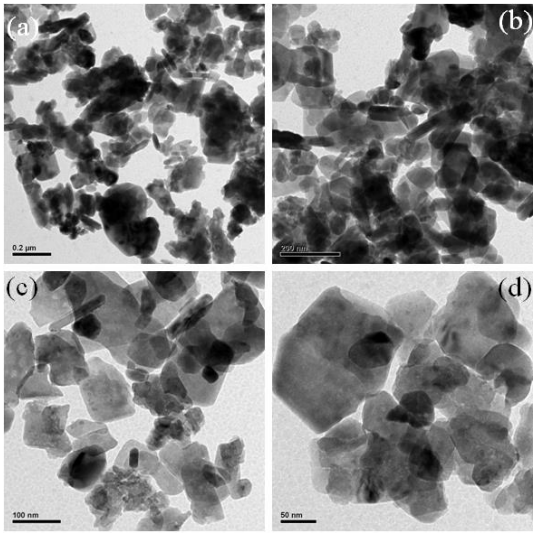

Fig. 4 (a-d) HR-TEM image of NiO nanoplates at different magnifications.

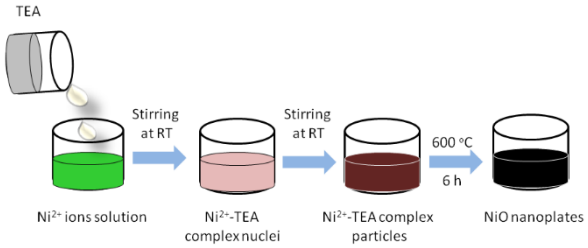

Fig. 5 Formation mechanism of NiO nanoplates

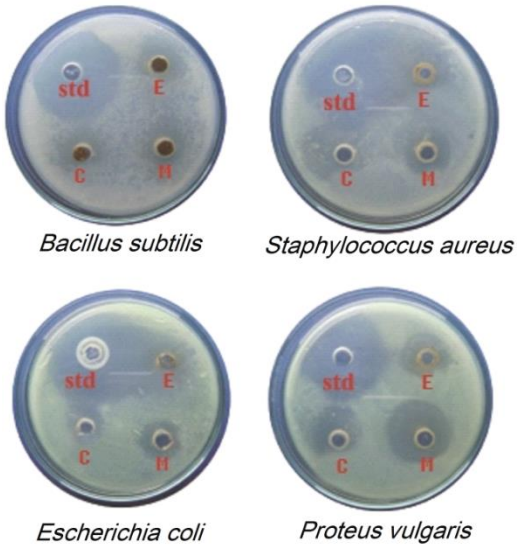

Fig. 6 Photographic images of an inhibition zone produced by NiO nanoplates in *B. subtilis, S. aureus, E. coli* and *P. vulgaris (Std –without solvent, E – ethanol, C-chloroform and M – methanol)*

3.3 Antibacterial Activity of NiO Powder

Fig. 6 shows the results of antibacterial activity of NiO nanoplates against *B. subtilis, S. aureus, E. coli and P. vulgaris* with different solvent such as ethanol (E), chloroform (C), and methanol (M). It can be seen that the NiO nanoplates exhibit maximum zone inhibition in without using any

solvent (Std). Maximum zone of inhibition was obtained in *B. subtilis, S. aureus,* and *P. vulgaris* with a zone diameter of 24 mm, 23 mm and 23 mm respectively. Comparatively, lowest zone of inhibition was observed in *E. coli* with a zone diameter of 19 mm. This result is comparable with other reports [16, 17]. The results reveal that NiO nanoplates show effective antibacterial activity. The growth inhibition of bacterial cells may be due to distractions of cell membrane by NiO nanoplates which results in breakdown of cell enzyme [18].

4. Conclusion

A simple and an inexpensive method has been reported to synthesis NiO nanoplates. The formation of NiO was confirmed by XRD and FTIR results. FE-SEM and HR-TEM images reveal the formation of plate-like NiO particles. Both XRD pattern and HR-TEM images showed that the NiO particles are in nanoscale. The antibacterial experiments show that the NiO nanoplates exhibit good zone of inhibition against *B. subtilis, S. aureus, E. coli* and *P. vulgaris*. The synthesized NiO nanoparticles can be also used in photocatalysis, smart windows, super capacitors etc.

References

[1] Tushar, R. Sathe, A. Agrawal, S. Nie, Mesoporous silica beads embedded with semiconductor quantum dots and iron oxide nanocrystals dual-function microcarriers for optical encoding and magnetic separation, Anal. Chem. 78 (2006) 5627-5632.

[2] A. Kalama, Abdullah, G.A. Sehemia, A.S. Alshihria, G. Dub, T. Ahmadc, Synthesis and characterization of NiO nanoparticles by thermal decomposition of nickel linoleate and their optical properties, Mater. Charact. 68 (2012) 77-81.

[3] F. Furlan, Ferreira, E. Avendan, Reversible electronic charge transfer between Au nanoparticles and electrochromic NiO matrices upon electrochemical cycling, J. Phys. Chem. C 111 (2007) 16608-16612.

[4] Y. Wang, J. Zhu, X. Yang, L. Lu, X. Wang, Preparation of NiO nanoparticles and their catalytic activity in the thermal decomposition of ammonium perchlorate, Thermochim. Acta 437 (2005) 106-109.

[5] E.L. Runnerstrom, A. Llordes, S.D. Lounis, D.J. Milliron, Nanostructured electrochromic smart windows traditional materials and NIR-selective plasmonic nanocrystals, Chem. Commun. 50 (2014) 10555-10572.

[6] S. Thota, Jitendrakumar, Sol–gel synthesis and anomalous magnetic behaviour of NiO nanoparticles, J. Phys. Chem. Solids 68 (2007) 1951-1964.

[7] C.L. Yuan, Room-temperature coercivity of Ni/NiO core/shell nanoparticles fabricated by pulsed laser deposition, J. Phys. Chem. C 114 (2010) 2124-2126.

[8] A. Barakat, M.A. Noaimi, M. Suleiman, A.S. Aldwayyan, B. Hammouti, T.B. Hadda, et al, One step synthesis of NiO Nanoparticles via solid-state thermal decomposition at low-temperature of novel aqua(2,9-dimethyl-1,10-phenanthroline) $NiCl_2$ complex , Int. J. Mol. Sci. 14 (2013) 23941-23954.

[9] S. Magaldi, S.M. Essayag, C. Hartung de capriles, C. Perez, M.T. Colella, C. Olaizola, Y. Ontiveros, Well diffusion for antifungal susceptibility testing, Int. J. Infect. Disease 8 (2004) 39-45.

[10] H. Qiao, Z. Wei, H. Yang, L. Zhu, X. Yan, Preparation and characterization of NiO nanoparticles by anodic arc plasma method, J. Nanomat. 2009 (2009) 795928.

[11] S.N. Anitha, I. Jayakumari, Synthesis and analysis of nanocrystalline $Fe_2Mn_2Ni_{0.5}Zn_{1.5}O_9$ at different treating temperatures, J. Nanosci. Tech. 1 (2015) 26-31.

[12] D. Khushalani, O. Dag, G.A. Ozin, A. Kuperman, Glycometallate surfactants Part 2: non-aqueous synthesis of mesoporous titanium, zirconium and niobium oxides, J. Mater. Chem. 9 (1999) 1491-1500.

[13] G.J. Li, S. Kawi, Synthesis, characterization and sensing application of novel semiconductor oxides, Talanta 45 (1998) 759-766.

[14] A.K. Ganguli, T. Ahmad, Nanorods of iron oxalate synthesized using reverse micelles: facile route for alpha-Fe_2O_3 and Fe_3O_4 nanoparticles, J. Nanosci. Nanotechnol. 7 (2007) 2029-2035.

[15] K. Giribabu, R. Suresh, R. Manigandan, E. Thirumal, A. Stephen, V. Narayanan, Aqueous based synthesis of Cu_5Se_4 nanosheets and characterization, J. Mater. Sci. Mater. Electron. 24 (2013) 1888-1894.

[16] J.D. Patel, U. Panchal, M. Panchal, B.A. Makwana, Green synthesis of silver nanoparticles using the leaf extracts and their microbial activity, J. Adv. Chem. Sci. 1 (2015) 82-85.

[17] A.S. Senina, A.A. Evdokimov, A.V. Moskvin, E.V. Fedorova, Synthesis, characterization and antimicrobial activity of amidrazone derivatives, J. Adv. Chem. Sci. 2 (2016) 183-187.

[18] S. Pal, Y.K. Tak, J.M. Song, Does the antibacterial activity of silver nanoparticles depend on the shape of the nanoparticle? A study of the gram-negative bacterium *Escherichia coli*, Appl. Environ. Microb. 73 (2007) 1712–1720.