Recent Research in Science and Technology 2012, 4(4): 50-53
ISSN: 2076-5061
Available Online: http://recent-science.com/

Synthesis and characterization of nickel oxide nanoparticles by self-propagating low temperature combustion method

Sharanabasava V.Ganachari[1], Ravishankar Bhat[1], Raghunandan Deshpande[2] and Venkataraman A*[1]

[1]Materials Chemistry Laboratory, Department of Materials Science, Gulbarga University, Gulbarga- 585 106 Karnataka, India
[2] H.K.E.'s Matoshree Taradevi Rampure Institute of Pharmaceutical Sciences, Sedam Road, Gulbarga- 585 105, Karnataka, India

Abstract
Nickel Oxide (NiO) nanoparticles have been synthesised by self-propagating low temperature Combustion synthesis method using Nickel salt with polyethylene glycol as fuel. As synthesized NiO nanoparticles was characterized by employing Fourier transform infrared spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and Energy dispersive X-ray microanalysis studies (EDAX) techniques and X-ray diffraction (XRD) confirmed the formation of NiO nanoparticles in the range 20 to 40 nm. SEM images clearly shows the NiO particles are in Nano size.

Keywords: NiO nanoparticles, X-ray Difraction, self-propagating low temperature combustion, Scanning Electron Microscope, Fourier transform infrared spectroscopy.

INTRODUCTION

Nanoparticles of transition metal oxides have been investigated by several workers in the last few years. Besides their structural aspects, magnetic properties of the oxide nanoparticles are of particular interest. Thus, it would be of value to know if the nanoparticles of antiferromagnetic oxides generally show evidence for ferromagnetic interaction at low temperatures, a behavior that has been reported by a few workers(R. H. Kodama et al 1999, S. A. Makhlouf et al 1997 & C. N. R. Rao and B. Raveau 1995). Nanoscale oxide particles of transition metals are gaining continuous importance for various applications such as catalysts, passive electronic components and ceramic materials (Patil K C et al 1997). Due to their small size, nanoparticles exhibit novel material properties that are significantly different from those of their bulk counterparts. Nickel oxide (NiO) nanoparticles with a uniform size and well dispersion are desirable for many applications in designing ceramic, magnetic, electro chromic and heterogeneous catalytic materials(Peterson M L et al 1997). Several researchers have prepared NiO nanoparticles by various methods like sol–gel (C N R Rao 1963), surfactant-mediated synthesis (C N R Rao 1994) thermal decomposition (Rao K J and Ramesh P D 1995), polymer-matrix assisted synthesis (Reddy Gopal G V et al 2000) and spray-pyrolysis (Smith J et al 1959). ultrasonic radiation, hydrothermal synthesis, carbonyl method, laser chemical method, pyrolysis by microwave, precipitation-calcination, micro emulsion method, and so on (Sverjensky D A 2003, Mallikarjuna N N and Venkataraman A 2003, Mallikarjuna N N et al 2003, Mallinson J C 1987,Venkataraman A et al 2001, Vijayanand H et al 2003). However, to the best of our knowledge, most of the reported experimental techniques for the synthesis of nanopowders are still limited in laboratory scale due to some unresolved problems, such as special conditions, tedious processes, complex apparatus, low yield, and high cost (Vijay A H 2000). From a useful viewpoint, it is vital to develop a way to manufacture high-quality nanopowders at high throughput with low cost (Yang B L and Kang H H 1982).

Current investigation is a self-propagating low temperature combustion route using nickel oxalate precursors for the synthesis of Nickel Oxide (NiO). Polyethylene glycol was used as a fuel for the precursor. In search of a suitable economic fuel, our use of Polyethylene glycol has given promising results in the conversion of precursor in to NiO.

The characterization study of as prepared NiO was undertaken by employing XRD, FTIR, FESEM and EDAX.

MATERIALS AND METHODS

Experimental

Nickel sulphate and oxalic acid used are AR grade chemicals. Polyethylene glycol of molecular weight 6,000 was obtained commercially (Merck Chemicals). The double distilled water is used is for preparation of solution.

Preparation of Nickel oxalate

The NiO is synthesised through self-propagating low temperature combustion route, employing nickel oxalate as precursor. This precursor is prepared by dissolving equimolar quantity of Nickel sulphate heptahydrate and oxalic acid in minimum amount of water. This mixture was well stirred in a three-necked flask. The Light green precipitate of nickel oxalate dihydrate obtained was filtered through sintered glass funnel and washed with double distilled water. Finally it was washed with dry acetone and dried under vacuum.

Received: Feb 12, 2012; Revised: March 18, 2012; Accepted: April 15, 2012.

*Corresponding Author

Venkataraman A
Materials Chemistry Laboratory, Department of Materials Science, Gulbarga University, Gulbarga- 585 106 Karnataka, India

Tel: +91-8472-263295; Fax: +91-8472-263206
Email: raman_chem@rediffmail.com

Synthesis of Nickel Oxide(NiO)

Thermal decomposition of Nickel Oxalate precursor with a fuel leads to the formation of high surface area NiO. The above prepared Nickel Oxalate was mixed with Polyethylene glycol (PEG) in the weight ratio 1:5 (Vijay *et al* 2000; Mallikarjuna *et al* 2003) and ground well in a pestle and mortar. The resultant solid was placed in a crucible and heated in air. It was observed that initially PEG melted, then frothed and finally ignited to give NiO as a residue. On cooling to room temperature, no traces of carbon impurities were observed in the final residue of NiO. As the reaction is fast, i.e. going to completion within 10 min, and ignites auto-catalytically, the exact temperature of the reaction could not be measured. However using a thermocouple the highest temperature of the reaction was found to be around 500°C.

Characterization techniques

The X-ray diffraction patterns were obtained employing a JEOL JDX-8p spectrometer using CuK radiation. The X-rays generator was operated at 30 kV. The scanning range, 2 was selected. The scanning speed = 1^0 min^{-1} were employed for precise lattice parameter determination. High purity silicon powder was used as an internal standard.

Fourier transformed infrared spectroscopy (FTIR) measurements carried out on a Perkin-Elmer spectrum one, instrument at a spectral resolution of 4 cm−1 in KBr pellets.

EDAX spectrum was obtained on an EDAX GENESIS4000 equipment at an accelerating voltage of 25.0 keV

The shape, size and distribution of the powder, as prepared tin oxide sample, microstructure of the sample have examined using a Leica-440 Cambridge Stereo scan, scanning electron microscope image. The SEM was operated at 20 kV. The samples were made conducting by the sputtering of gold using a Poloron DC "sputtering unit" operated at 1.4 kV and 18-20 mA.

RESULTS AND DISCUSSION

X-ray diffraction study (XRD)

The purity and crystallinity of the as-synthesized NiO nanoparticles were examined by using powder X-ray diffraction (XRD) as shown in Figure 1. It can be seen from Figure 1 that the diffraction peaks are low and broad due to the small size effect and incomplete inner structure of the particle. The peaks positions appearing at 2θ is 37.21◦, 43.22◦, 63.10◦, 75.20◦, and 79.39◦ can be readily indexed as (101), (012), (110), (113), and (006) crystal planes of the bulk NiO, respectively. All these diffraction peaks can be perfectly indexed to the face-centered cubic (FCC) crystalline structure of NiO, not only in peak position, but also in their relative intensity of the characteristic peaks, which is in accordance with that of the standard spectrum (JCPDS, No. 04-0835). The XRD pattern shows that the samples are single phase and no any other impurities distinct diffraction peak except the characteristic peaks of FCC phase NiO was detected. This result shows that the physical phases of the NiO nanoparticles have higher purity prepared in this work. The NiO lattice constant calculated from the XRD data is 4.1729 A° , which is in good agreement with the reported data.

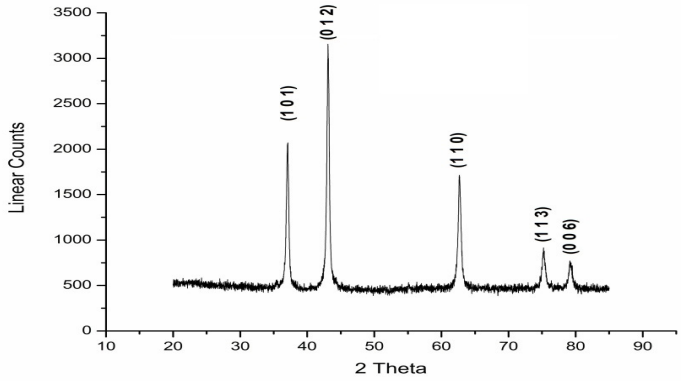

Fig 1.

This is again confirmation with FTIR and EDX studies, where additional frequencies are observed in FTIR spectrum and Ni peaks are observed in EDX pattern. The details XRD patterns of the NiO nanoparticles are shown in fig 1. All the reflection peaks with relative intensities of different planes, indexed in the figure, specify the presence of NiO. The sharpness and the intensity of the peaks indicate the well crystalline nature of the prepared sample.

FTIR study

Figure 2 shows the FTIR spectra of NiO nanoparticles, which showed several significant absorption peaks. The broad absorption band in the region of 600–700 cm−1 is assigned to Ni–O stretching vibration mode; the broadness of the absorption band indicates that the NiO powders are nanocrystals. The size of samples used in this study was much less than the bulks form NiO, so that NiO nanoparticles had its FTIR peak of Ni–O stretching vibration and shifted to blue direction. Due to their quantum size effect and spherical nanostructures, the FTIR absorption of NiO nanoparticles is blue-shifted compared to that of the bulk form. Besides the Ni–O vibration, it could be seen from Figure 2 that the broad absorption band centered at 3440 cm−1 is attributable to the band O–H stretching vibrations and the weak band near 1635 cm−1 is assigned to H–O–H bending vibrations mode were also presented due to the adsorption of water in air when FTIR sample disks were prepared in an open air. These observations provided the evidence to the effect of hydration in the structure. Meanwhile, it implied the presence of hydroxyl in the precursor, and the broad absorption around 767 cm−1 is assigned to the band C=O stretching vibrations. The serrated absorption bands in the region of 1000– 1500 cm−1 are assigned to the O–C=O symmetric and asymmetric stretching vibrations and the C–O stretching vibration, but the intensity of the

band has weakened, which indicated that the ultrafine powers tend to strong physically absorption to H2O and CO2.

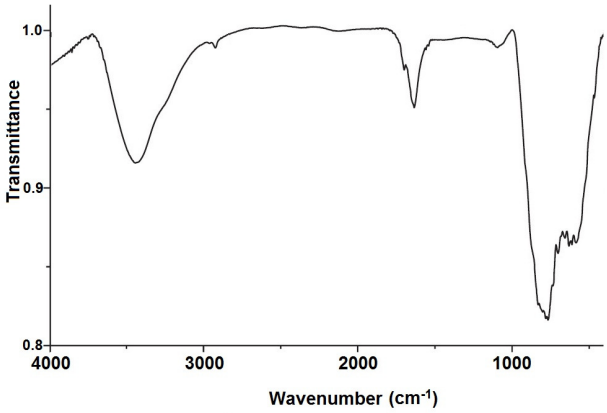

Fig 2.

Energy dispersive X-ray microanalysis studies (EDAX)

The energy dispersive X-ray microanalysis was carried to know the presence of Nickel oxide. Figure 3 shows the EDX pattern of NiO sample. This pattern shows the presence of both Nickel and Oxygen peaks.

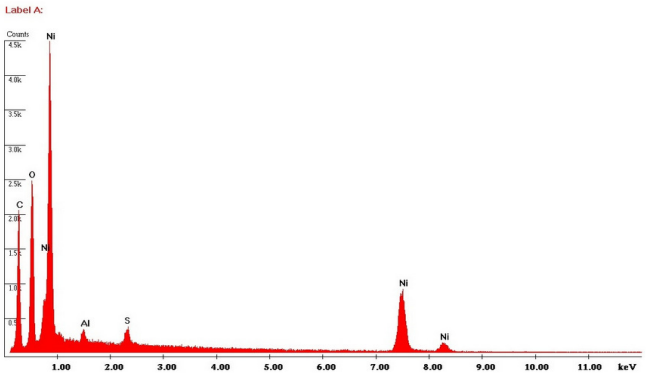

Fig 3.

Scanning Electron Microscope study (SEM)

Morphology of NiO nanoparticles was studied by scanning electron microscope (SEM) tool. Figure 4 (a-b) shows the particle morphology of NiO at low (a) and high (b) resolution. The particles are mostly irregular spherical shape with a nanosize range 20-40 nm. Some particles are found as agglomerated surface are observed.

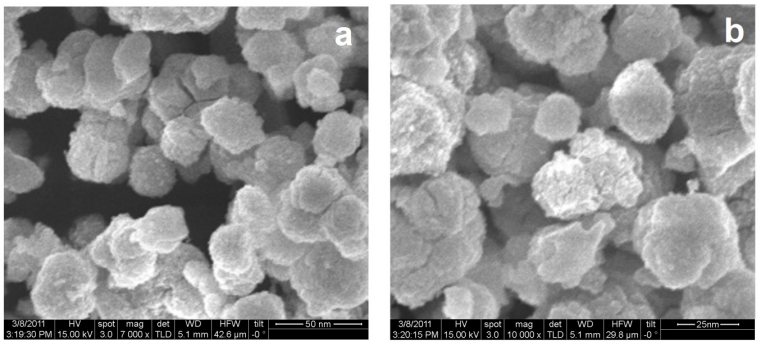

Fig 4.

CONCLUSION

Nanosized NiO particles were synthesized using self-propagating low-temperature combustion route because of its simplicity and easy scale up. The results obtained from crystallite size from XRD and SEM images confirm the nanocrystalline nature of the synthesized products. The results suggest that the self-propagating low-temperature combustion method is an effective pathway for producing high-quality NiO nanosized powder.

ACKNOWLEDGEMENT

Authors are grateful to UGC, Major Research Project (F. No. 33-307/2007(SR) and DAE-BRNS Project (No.2009/34/14/BRNS),

for financial assistances.

REFERENCES

[1]R. H. Kodama, 1999. *J. Magn. Magn. Mater*., 200:359.

[2]S. A. Makhlouf, F. T. Parker, F. E. Spada and A. E. Berkowitz,1997. *J. Appl. Phys.,* 81, 5561.

[3]C. N. R. Rao and B. Raveau, 1995.Transition Metal Oxides, 2nd edn, Wiley-VCH, Germany.

[4]Patil K C, Aruna S T and Ekambaran S 1997. *Curr. Opp. Solid State Mater. Sci.* 2:158

[5]Peterson M L, White A F, Brown G E, Jr Parks G A, 1997. *Environmental Sci. Technol*. 31:1573

[6]Rao C N R 1963 *Chemical applications of infrared spectroscopy* (New York & London: Academic Press)

[7]Rao C N R, 1994. *Chemical approaches to the Synthesis of inorganic materials* (New Delhi: Wiley Eastern Ltd.)

[8]Rao K J and Ramesh P D, 1995. *Bull. Mater. Sci.* 18:447

[9]Reddy Gopal G V, Sheela Kalyana and Manorama S V, 2000. *Int .J. Inorg. Mater.* 2:301

[10] Smith J, Wijn H P T, Phillips N N and Gloeilampenjabricken Eindnoven, 1959. *Ferrites* (Holland) p.144A

[11] Sverjensky D A ,2003. *Nature* 364:776

[12] Mallikarjuna N N and Venkataraman A ,2003. *Talanta* 60:147

[13] Mallikarjuna N N, Govindraj B, Lagashetty Arunkumar and Venkataraman A, 2003. *J. Therm. Anal. Cal.* 71:915

[14] Mallinson J C 1987 *The foundations of magnetic recording* (San Diego: Academic Press)

[15] Venkataraman A, Hiremath V A, Date S K and Kulkarni S M 2001 *Bull. Mater. Sci.* 24(6):101

[16] Vijayanand H, Lagashetty Arunkumar, Mallikarjuna N N and Venkataraman A. 2003, *Asian J. Chem.*15:79

[17] Vijay A H 2000 *Synthesis and Studies of Gamma Ferric Oxide* (Ph.D. Thesis, Gulbarga University, Gulbarga, India)

[18] Yang B L and Kang H H 1982, *J. Catal.* 77: 410