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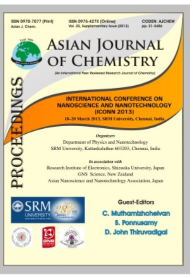

Facile Synthesis of Nickel Oxide Nanoparticles and Their Structural, Optical and Magnetic Properties†

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Nickel oxide nanoparticles have been successfully synthesized through microwave assisted reaction of nickel nitrate, sodium hydroxide and citric acid as a capping agent. The structure, morphology, optical and magnetic properties of the NiO products are characterized by XRD, TEM, FTIR, UV-VIS and VSM. XRD results revealed that the resultant sample showed cubic structure with average grain size less than 25 nm. Analysis of FTIR spectra confirmed the composition of the product and the existence of nickel oxide vibration. TEM image demonstrated that the obtained nickel oxide nanoparticles with size less than 25 nm. The optical absorption band gap of nanocrystalline NiO was estimated to be 3.21 eV. Magnetic measurement showed that the nickel oxide nanoparticles exhibit ferromagnetic behaviour at room temperature (300 K). Based on experimental observation and analysis, a possible mechanism is proposed to enrich the properties of NiO nanostructured material for various applications such as storage devices, gas sensors, *etc*.

Key Words: Microwave, Nanoparticles, Ferromagnetic, Saturation.

INTRODUCTION

Magnetic nanoparticles are of great interest because they exhibit interesting optical, electronic, magnetic and chemical properties. Among the magnetic nanoparticles, antiferromagnetic nanoparticles (AFN) are promising candidates for the recording/reading of the audio and video signals and storage devices such as magnetic tapes, magnetic random access memory, floppy disks and hard disks *etc*. Nickel oxide is one of the most versatile magnetic materials among transition metal oxides exhibits antiferromagnetic nature with Neel temperature of 523 K[1]. In recent reports of NiO materials, investigation of magnetic properties for NiO nanoparticles have attained considerable interest to improve the magnetic performance of NiO nanostructures and utilized for storage devices and other applications.

A wide variety of methods are adopted for the preparation of nickel oxide nanoparticles such as sol-gel, chemical precipitation, microwave assisted method and anodic arc plasma technique. Microwave assisted method have recently been acknowledged as a promising route for preparation of nickel oxide nanocrystalline materials[2]. In the present study, we successfully synthesized NiO nanoparticles by efficient and shorter heating rate of microwave assisted reaction with the addition of capping agent (citric acid). The structural, functional, morphological, optical and magnetic properties were studied and analyzed using X-ray diffraction, Fourier transform infrared, transmission electron microscope, UV-visible spectroscopy and vibration sample magnetometer. The obtained result implies that the role of capping agent (citric acid) in the solution produced nano sized particles because of its effectiveness which exhibits 0.8 emu/g saturation magnetization which is in well agreement with the previous reports.

EXPERIMENTAL

In a typical experiment, 0.5 M of $Ni(NO_3)_2{\cdot}6H_2O$ was dissolved in 50 mL distilled water. Then 1 g of citric acid was added to the above solution under vigorous stirring, followed by an addition of 20 mL of 2 M NaOH solution drop wise to form a clear blue solution at pH 9. Then, the solution was transferred to autoclave bottle. The container is ready for microwave treatment (microwave oven Samsung CE1031LFB) which works with a frequency of 2.45 GHz having a maximum microwave power of 900 W. The solution was irradiated by microwave with the power of 300 wattage of irradiation for 15 min. The obtained green precipitates were collected by centrifugation and the precipitates were rinsed with distilled water to remove soluble ions and impurities. The precipitate

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was dried at 110 ºC in a hot air oven for 12 h to get the sample of Ni(OH)$_2$ architectures. Then, the as-prepared powder was calcined under air at 400 ºC for 2 h to obtain NiO nanoparticles. The NiO nanoparticles synthesized using citric acid is denoted as NiO-CA.

RESULTS AND DISCUSSION

Structural analysis: Fig. 1 shows the XRD pattern of NiO nanoparticles calcined at 400 ºC for NiO-CA sample. The obtained NiO sample could be indexed to cubic NiO (FCC phase) with lattice parameter a = 4.194 Å and matched with standard JCPDS card no 89-7130. All the diffraction peaks of NiO samples exhibits standard peaks at 2θ values 37º, 43º, 63º, 75º and 79º corresponds to (111), (200), (220), (311) and (222) planes respectively[3]. The absence of any other peaks in the XRD pattern illustrates the purity of nanostructures. The average grain size were calculated from Debye-Scherrer formula

$$D = 0.9\lambda/\beta \cos \theta$$

where λ is the X-ray wavelength, β is the full width at half maximum intensity and θ is the Bragg's angle. The average grain size of NiO-CA nanoparticles is 25 nm because of the effectiveness of citric acid in the solution.

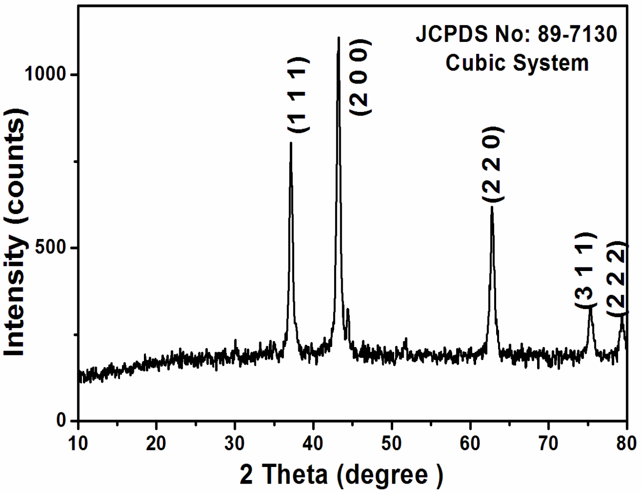

Fig. 1. X-ray diffraction pattern for NiO-CA nanoparticles

Functional analysis: FTIR spectroscopy is a useful tool to investigate functional groups, modes of vibration and behaviour of nickel precursor. Fig. 2 shows the FT-IR spectrum of NiO-CA sample. The absorption band appears around 3400-3580 cm^{-1} and 645 cm^{-1} are due to the absorption of OH stretching mode and the band exists near 1660-1600 cm^{-1} (H-O-H bending mode) is related to the evidence of water absorption in the NiO product. The characteristic stretching band NaNO$_3$ and NO$_3$ are observed around 2427, 1388 and 825 cm^{-1}. It is important to note that, IR spectrum for NiO-CA showed strong band exists at 431 cm^{-1} is the nature of vibration modes of NiO [4 and 5].

Morphological analysis: Fig. 3(a-c) represents the TEM images of NiO nanoparticles for NiO-CA. The average size of the particles was found from the TEM analysis. Citric acid used NiO samples produced large number of particles with less agglomerated nanoparticles. The higher magnification TEM images (50 and 20 nm scale) explained the detailed information of particle sizes of NiO sample. In this magnified view, the average particle size was found to be 20-25 nm with weakly agglomerated nanoparticles for citric acid assisted NiO nanostructures. From these discussions, one can say that average particle size observed from TEM images was around 25 nm which is more comparable to the average size calculated by Debye-Scherer formula from XRD data.

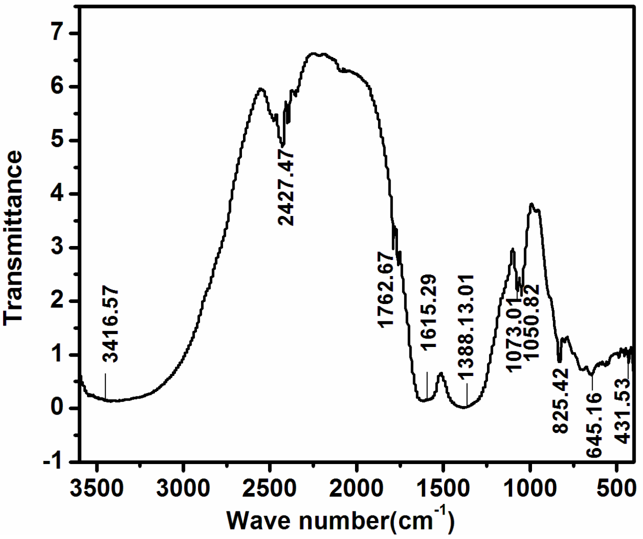

Fig. 2. FTIR spectrum for NiO-CA nanoparticles

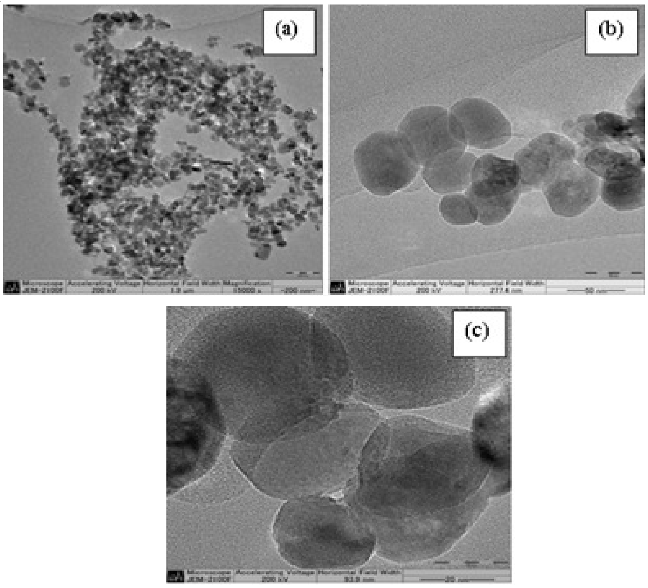

Fig. 3. TEM images of NiO-CA nanoparticles

Optical analysis (UV-VIS): Fig. 4 shows the optical absorption spectra for NiO-CA. The entire NiO nanoparticles exhibit the characteristic absorption edge in the UV region observed at 307 nm for NiO-CA. This absorption in UV region is the band gap absorption of NiO nanostructures. The band gap of NiO has been determined by the optical absorption spectrum using the following equation:

$$(\alpha h\nu)^n = B(h\nu - E_g)$$

where α is the absorption co-efficient, hν is the photon energy, n = 2 for direct transition and B is the constant related to the material. The extrapolation of $(\alpha h\nu)^2$ *versus* hν graph gives the band gap value of NiO. The band gap value of NiO nanoparticles for NiO-CA is 3.21 eV which is lower when compared to bulk NiO (4.0 eV). This lower band gap value of NiO nanoparticles exhibits the nature of blue shift due to quantum confinement effect in the nanoscale region[6].

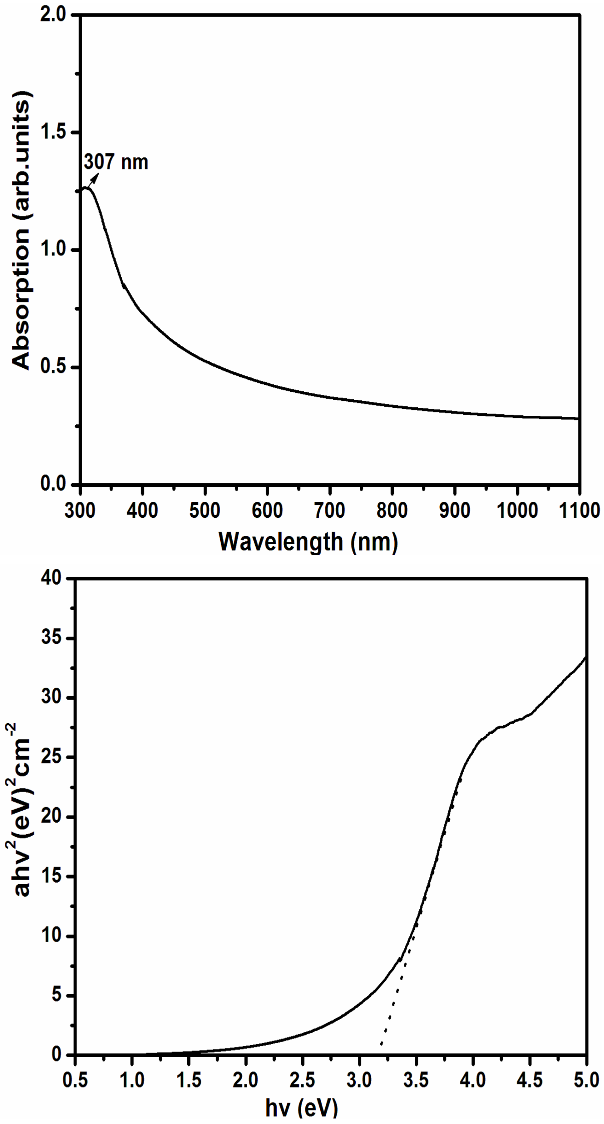

Fig. 4. Optical absorption and band gap curve for NiO-CA nanoparticles

Room temperature magnetic analysis (VSM): Fig. 5 shows the hysteresis loop for NiO-CA nanoparticles at room temperature magnetic measurement carried out by using vibrating sample magnetometer. The magnetic property of NiO-CA sample showed the ferromagnetic nature of NiO nanostructures. It is shown that NiO-CA nanoparticles produces higher value of saturation magnetization (0.8 emu/g) with the smallest particle size. It has been strongly concluded that the magnetic behaviour of NiO nanoparticles depends upon the size of nanoparticles. The present study revealed that NiO-CA sample produces narrow sized hysteresis loop with saturation magnetization of 0.8 emu/g, which is in good agreement with the literature report[7].

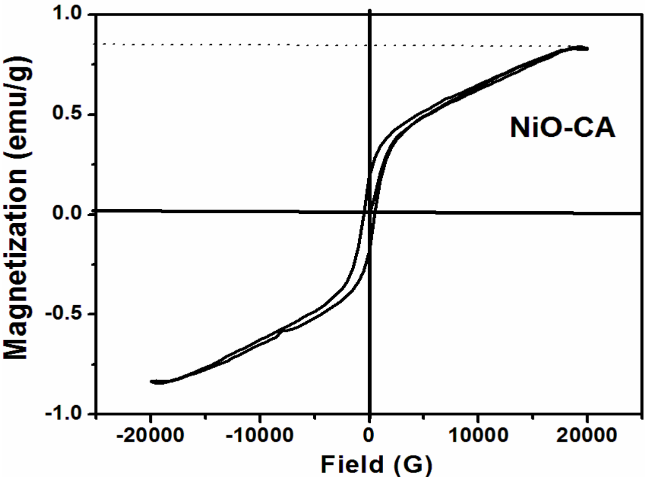

Fig. 5. Room temperature hysteresis loop for NiO-CA nanoparticles

Conclusion

Cubic phase of NiO nanoparticles with an average size of 25 nm was obtained by microwave synthesis method. The microstructural parameters were obtained using X-Ray diffraction pattern. The observed TEM images demonstrated that NiO-CA produces nanoparticles having average size of 25 nm which is more comparable to the calculated value through XRD data. NiO nanoparticles showed lower band gap of 3.21eV compared to bulk NiO because of the effect of quantum confinement. Room temperature magnetic measurement displayed ferromagnetic nature of NiO nanoparticles with saturation magnetization of 0.8 emu/g. All the above results suggest that NiO-CA nanoparticles have a really appreciable magnetic response and are suitable magnetic material for magnetic devices.

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