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Wet chemical synthesis and characterization of NiO nanoparticles

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ABSTRACT

The NiO nanoparticles were successfully synthesized by wet chemical method. The products were calcinated at 600°C and it was characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), energy dispersive X-ray spectroscopy (EDAX), Fourier transform infrared spectroscopy (FTIR) and UV–vis absorption spectroscopy. The results obtained confirm the presence of nickel oxide nano powders produced during chemical precipitation.

Key words: Nickel oxide, wet chemical method, nanoparticles, X-ray diffraction studies, tunneling electron microscopy

INTRODUCTION

In the recent years, the different size and various shapes of nanomaterials have been realized through a wet-chemical synthesis because of its wide range of application. Owing to this reason, researchers focusing an increasing interest to fabricate nanostructured materials [1]. In this present work, we prepared nano particles by using wet-chemical synthesis [2]. Its applications are extended by changing its physical and chemical properties under nanoscale due

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to large range of surface to volume ratio [3]. The chosen metal oxide nanoparticles like nickel oxide is an important transition metal oxides of p-type semiconducting material with a band gap of 4.0 eV [4,5]. These metal oxide nanoparticles were used as gas sensors, electro chromic films and fuel cells etc. Generally, nanocrystalline metal oxides have been prepared by wet-chemical techniques such as sol-gel, solvothermal and co-precipitation methods etc., [6]. Here we prepared NiO nanoparticles by using co-precipitation method.

MATERIALS AND METHODS

Materials used

Nickel nitrate hexahydrate [$Ni(NO_3)_2.6H_2O$] and sodium hydroxide [NaOH] were from Merck (Mumbai, India) and deionized water was used for the preparation of solution. The absolute ethanol was used for the washing of final product.

Experimental Method

In this method, 0.1M $Ni(NO_3)_2.6H_2O$ solution was prepared by using double distilled water under constant stirring. While at room temperature, 0.8M NaOH solution was added drop by drop. This solution was stirred constantly at a temperature of 80±5 °C for 4 hours. After completion of the reaction, the green coloured precipitate was obtained and it was thoroughly washed with double distilled water to remove all the other ions and then centrifuged. The final precipitate was dried in a hot air oven at 80°Cfor 2 hours. The above resulting dried precursors was crushed into powder and calcined at 600 °C for 5 hours. The final product (NiO) was stored in air tight container for further analysis.

Characterization of NiO NPs

The phase purity of the synthesized NPs was determined by X-ray diffractometer (model: X'PERT PRO PAN analytical). The morphological features of the sample were measured by Transmission electron microscopy (model: Tecnai F20) and subjected to TEM-EDS for high speed elemental analysis. The vibrational frequency was measured by Fourier transform infra-red spectroscopy (Perkin Elemer). The absorption spectrum of the sample was measured on a Perkin Elmer (Lambda 35).

RESULTS AND DISCUSSION

The phase purity of the sample was characterized through XRD studies. Figure 1 depicts the XRD pattern of the as synthesized NiO nanoparticles. The XRD peaks are appeared at angles (2θ) of 37.26˚, 43.50˚, 63.08˚, 75.39˚ and 79.50˚ corresponding to (111), (200), (220), (311), and (222) planes, and very good accordance with a cubic NiO crystal structure (JCPDS Card No: 78-0643: space group = Fm3/m).The grain size of the crystallites of as synthesized product was calculated using Debye-Scherrer formula.

$$D = \frac{0.9\lambda}{\beta\cos\theta} \text{ ---------- (1)}$$

Where λ is the wavelength of X-ray used (0.15418nm in the present case), β is the full width in radiation at half-maximum of the peak, and θ is the Bragg angle of X-ray diffraction peak. The average crystallite size of the as synthesized nanoparticles was 21.49 nm.

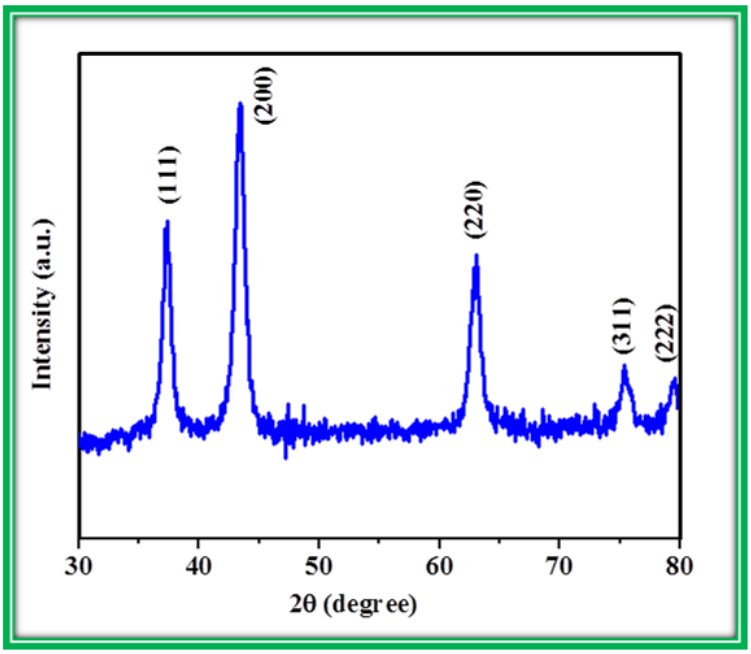

Figure 1: XRD pattern of NiO Nanoparticles

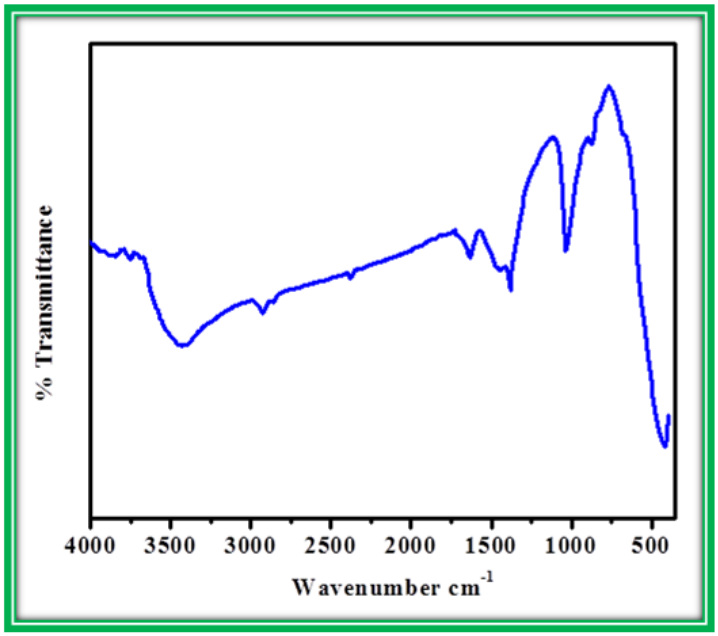

Figure 2: FTIR spectrum of NiO nanoparticles

Figure 2 displays the FTIR spectrum of NiO nanoparticles. The broad absorption band at 3425 cm^{-1} and the weak peak at 1627 cm^{-1} are assigned to OH stretching and bending modes of water, respectively [7]. The wide absorption band around 1381 cm^{-1} can be ascribed to CO_3^{2-} ions. The peak at 1033 cm^{-1} corresponds to stretching and bending vibrations of the intercalated C-O species [8] and the bands at 2924 cm^{-1} and 2854 cm^{-1} can be assigned to CH_2 vibrations. The peak observed at 416 cm^{-1}, which correspond to Ni-O nanoparticles stretching mode [9].

Figure 3(a) demonstrates the UV-visible spectrum of the NiO nanoparticles suspension as obtained by ultrasonic dispersion in water. A strong absorption peak in the UV region is observed at wavelength of 272 nm.

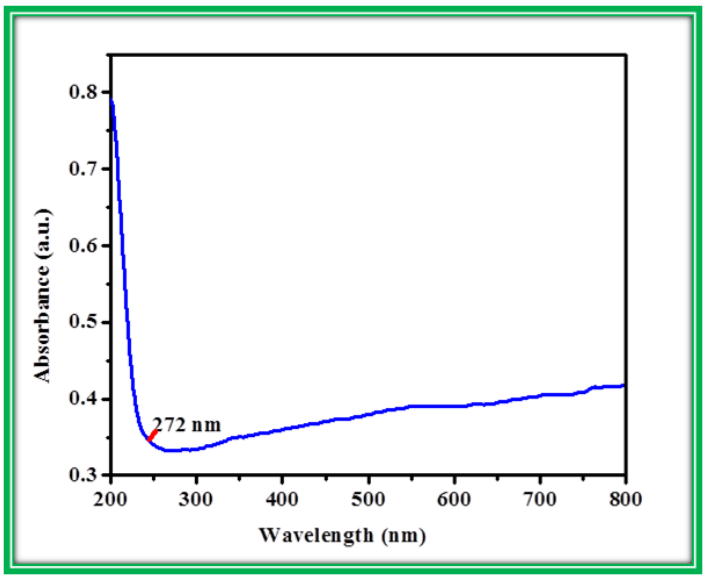

Figure 3 (a): UV-Visible Spectrum of NiO Nanoparticles

This absorption in the UV region is attributed to band gap absorption of NiO [10]. It is due to interaction predicts that the top of the valence band consists of the oxygen 2p band and the bottom of the conduction band is mainly derived from Ni 3d states [11]. Figure 3(b) displays the band gap spectrum of NiO nanoparticles. The absorption band gap (E_g) is usually achieved with the aid of the following equation.

$$\alpha = \frac{k\,(h\upsilon - E_g)^{n/2}}{h\upsilon} \text{---------- (3)}$$

Where k is a constant, E_g is the band gap and n is a constant equal to 1 for direct gap semiconductors and 4 for indirect band gap semiconductors materials.

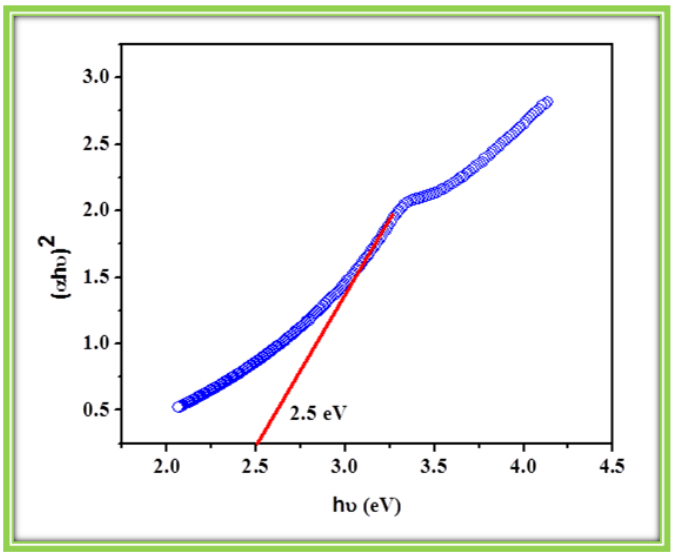

Figure 3 (b): Band Spectrum of NiO Nanoparticles

The variation of $(\alpha h\upsilon)^2$ versus $h\upsilon$ is linear at the absorption edge which confirmed direct band gap transition in NiO. The band-gap energy of the as-prepared NiO nanoparticles is found to be 2.5 eV, which is smaller than the bulk NiO (4.0 eV) [12].

Figure 4 shows the TEM image of synthesized NiO nanoparticles. From the figure, the surface is observed to be smooth and covered with uniform spherically shaped grains and also we observed the agglomerated particles due to surfactant free synthesis of NiO nanoparticles.

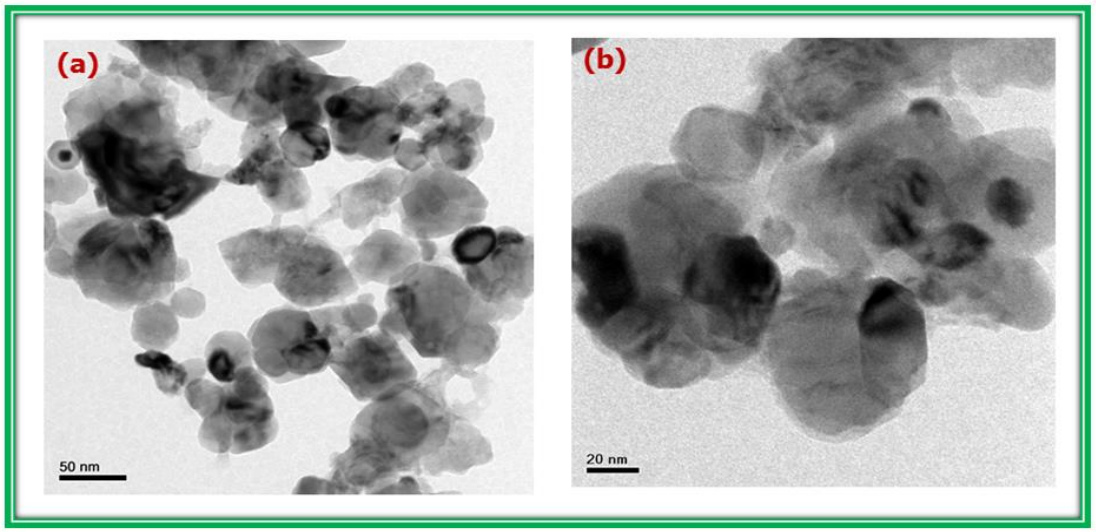

Figure 4: TEM images of NiO nanoparticles

The grains are distributed uniformly over the entire surface of the Nanoparticles. The sizes of grains are found to be in the range between 21 nm and 30 nm. The average size of the grains is found to be 21 nm and it is in good agreement with the XRD results.

Figure 5 shows typical EDAX pattern of the NiO nanoparticles. The EDAX data clearly indicates that the elements present in the sample are 50:50 ratio (Weight percentage: Ni = 50.20, O = 49.80). The elemental analysis confirmed that only Ni and O are present in the synthesized sample and there was no impurities present in the sample.

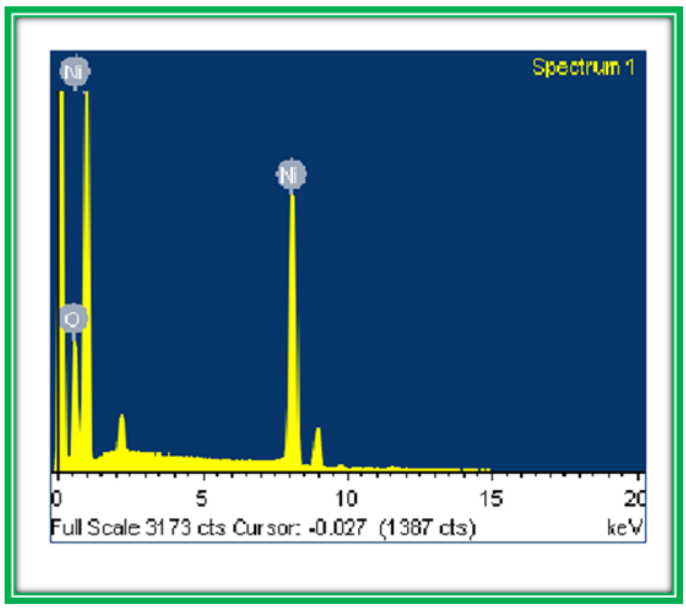

Figure 5: EDAX pattern of NiO nanoparticles

CONCLUSION

Nearly spherical nanoparticles (21-30 nm) of NiO have been synthesized by adopting co-precipitation method. The XRD study has confirmed the formation of NiO nanoparticles with cubic structure with a mean size of 21 nm. The grains size of NiO nanoparticles is in close agreement with TEM and XRD studies. FTIR result evidenced that the NiO nanoparticles formed from the decomposition of $Ni(OH)_2$. The EDAX analysis confirmed that the purity of the synthesized NiO nanoparticles. The UV-Vis spectrum showed the optical band gap of 2.5 eV, which indicates the red shift on the size reduction.

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