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Research Paper

Sol-gel combustion and characterization studies of nickel oxide nanoparticles using microwave synthesis

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Abstract

Semiconductor NiO nanoparticles were successfully synthesized by a simple sol-gel combustion method using urea as the fuel. The prepared NiO nanoparticles were characterized by powder XRD pattern, SEM, TEM, EDX analysis and VSM analysis. Powder XRD results were confirmed the pure form of cubic NiO phase. Surface morphology of the NiO nanoparticles was analyzed by SEM and TEM analysis, and confirmed the particle like nano-crystal morphology. of the samples were investigated by room temperature VSM and their magnetic - hysteresis (M-H) loop confirmed the super paramagnetic behavior of the NiO nanoparticles.

Keywords: NiO; Nanoparticles; Combustion; Electron microscopy; Magnetic properties.

1. Introduction

Nowadays, nano-structured metal oxides exhibit unique properties, which are considerably different from their same bulk materials [1-5]. It well known that the physico-chemical properties of the metal oxide nanocrystals mainly depend on their microstructure, surface area, and the presence of dopant cations [6-10]. Among the metal oxide nanoparticles, nickel oxide (NiO), exhibit excellent electronic, opto-magnetic and catalytic properties [11]. NiO

is a p-type semiconductor of wide band gap (3.6–4.0 eV) [12-15]. Due to the excellent physico-chemical properties, it was recommended for the applications of gas sensor, photo-catalyst, battery, fuel cells and also dye sensitized solar cells [16-20].

A variety of synthesis methods including sol-gel, polyol, solvothermal, combustion and co-precipitation [21-25] methods, have been used to synthesize of metal oxide nanoparticles. But the above said approaches meet some disadvantageous such as high-energy consuming, needed costly equipment and require rather long reaction times. Among the above methods, combustion method using glycine as the fuel has more advantages, such as one-pot process, high purity products and low cost. Newly, many oxide and sulphide have been synthesized by this combustion process [26-35]. From this method, nano-sized particles with larger surface area of the samples obtained. In this present study, the authors have synthesized NiO nanoparticles by a simple combustion method and investigated their structural, vibtrational, morphological and magnetic properties. The samples were characterized by powder XRD, SEM, TEM and VSM analysis.

2. Experimental part

2.1 Materials and methods

All the reagents used were analar grade obtained from Merck, India and were used as received without further purification. The method rely on the advantage of the propellant chemistry in constructing the redox mixtures, in which metal nitrate acts as an oxidizing reactant and fuel glycine as a reducing reactant. Stoichiometric amounts of nickel nitrate and glycine were dissolved separately in 10 ml of de-ionized water and poured into a silica crucible and stirred for 15 minutes to obtain clear solution. The solution treated in an air furnace at 500°C for 2 h at a heating rate of 5°C/min, and cooled at the same rate. Initially, the solution boiled and underwent dehydration followed by decomposition with the evolution of gases and became a solid. The obtained solid was washed well with water and followed by ethanol and dried in a hot air oven at 70°C for 2 h. The prepared samples were used for further characterizations.

2.2. Characterizations

The structural analysis was performed using a Philips X'pert X-ray diffractometer with CuKα radiation at λ = 1.540 Å. Morphological studies and energy dispersive X-ray analysis was

performed with a Jeol JSM6360 high resolution scanning electron microscope. The transmission electron micrographs were carried out by Philips - TEM (CM20). The magnetic properties were investigated using vibrating sample magnetometer (VSM) at room temperature in an applied magnetic field sweeping from -10 to +10 KOe using a PMC MicroMag 3900 model vibrating sample magnetometer (VSM) equipped with 1 Tesla magnet.

3. Results and discussion

3.1. Structural analysis

The structural and crystal phase of NiO nanoparticle was determined by X-ray powder diffraction pattern. Fig. 1 shows the powder XRD pattern of NiO nanoparticles. The diffraction peaks located at 37.38, 43.37, 62.94, 75.48, and 79.48º, corresponding to (111), (200), (220), (311) and (222) *hkl* planes, which confirmed the formation of a pure cubic NiO phase with JCPDS No. 78-0643 [10].

The average crystallite size was calculated using Scherrer's formula given in Eq. (1)

$$L = \frac{0.89\lambda}{\beta \cos\theta} \qquad \text{---- (1)}$$

where L is the crystallite size, λ, the X-ray wavelength, θ, the Bragg diffraction angle and β, the full width at half maximum (FWHM). The average crystallite size 'L' calculated from the diffraction peaks was found to be around 32.15 nm. The lattice parameters were calculated using the formula given in Eq. (2):

$$\sin^2\theta = \frac{\lambda^2}{4}\left[\frac{4}{3}\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{l^2}{c^2}\right] \qquad \text{---- (2)}$$

where θ is the diffraction angle, λ, the incident wavelength (λ = 1.542 Å), h, k, and l are Miller's indices. The calculated lattice parameter value is to be a = 5.116 Å and c= 13.442 Å. The calculated values are consistent with the reported values (a = 5.031 Å and c = 13.653 Å, JCPDS No. 89-2598).

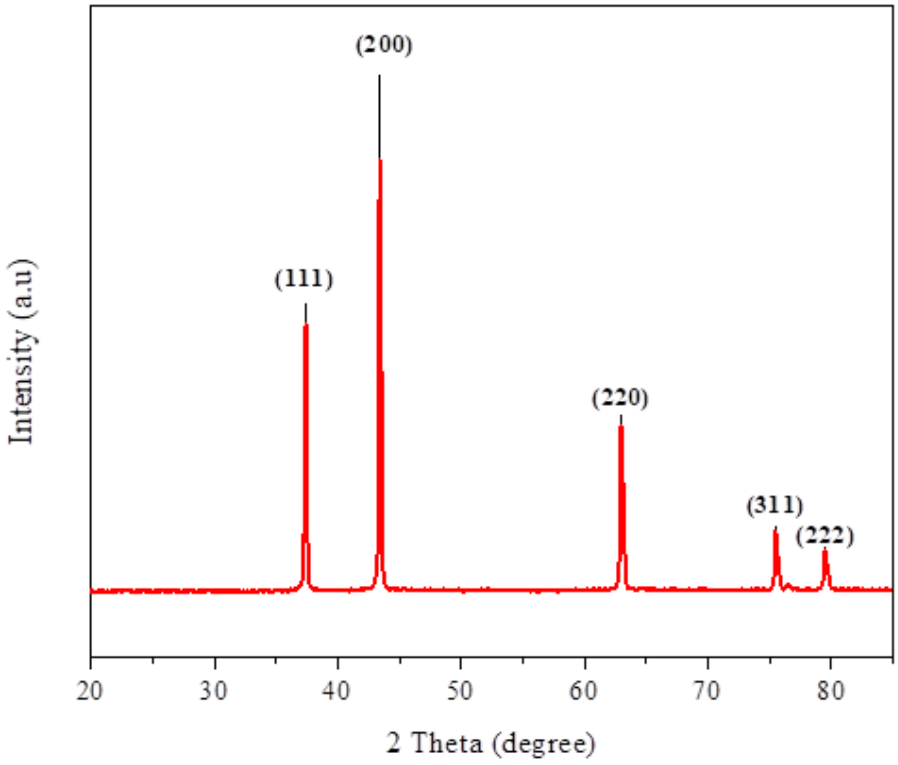

Fig. 1. Powder XRD patterns of NiO nanoparticles.

3.2. FT-IR analysis

Functional groups of the samples was confirmed by Fourier transform infra-red (FT-IR) analysis. Fig. 2 shows the FT-IR spectra of NiO nanoparticles. It is well known that the metal oxygen (M-O) bands are appeared in the range of 460 – 950 cm^{-1}. A band at the range of 565 and 725 cm^{-1} is assigned to Ni-O band stretching. However, the bands observed at ~ 3445, ~1385 and ~ 1625 cm^{-1} prove the presence of adsorbed water on the surface.

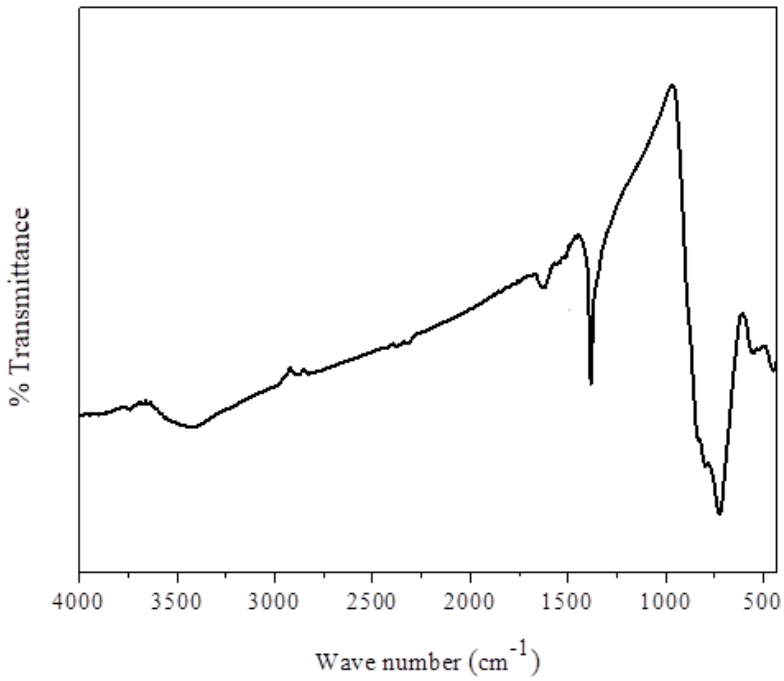

Fig. 2. FT-IR spectra of NiO nanoparticles.

3.3. SEM analysis

Fig. 3a and b shows the SEM image and EDX spectra of NiO nanoparticles. Fig. 2a shows the presence of flakes – like nanoparticles morphology with smaller agglomeration. Therefore, we can infer that the agglomerated NiO nano-crystals have been formed during the combustion process. The elemental composition was analyzed by energy dispersive X-ray analysis (EDX) as shown in Fig. 3b. The EDX results showed the presence of Ni and O by the appearance of Ni and O peaks without any other characteristic peaks are indeed free from other impurities.

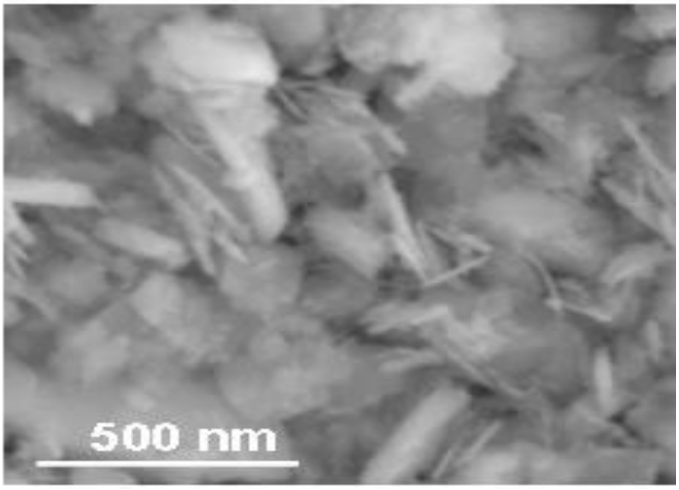

Fig. 3. SEM image of NiO nanoparticles.

3.4. EDX analysis

Fig. 4 shows the EDX spectra of NiO nanoparticles. The elemental composition was analyzed by energy dispersive X-ray analysis (EDX) as shown in Fig. 4. The EDX results showed the presence of Ni and O by the appearance of Ni and O peaks without any other characteristic peaks are indeed free from other impurities.

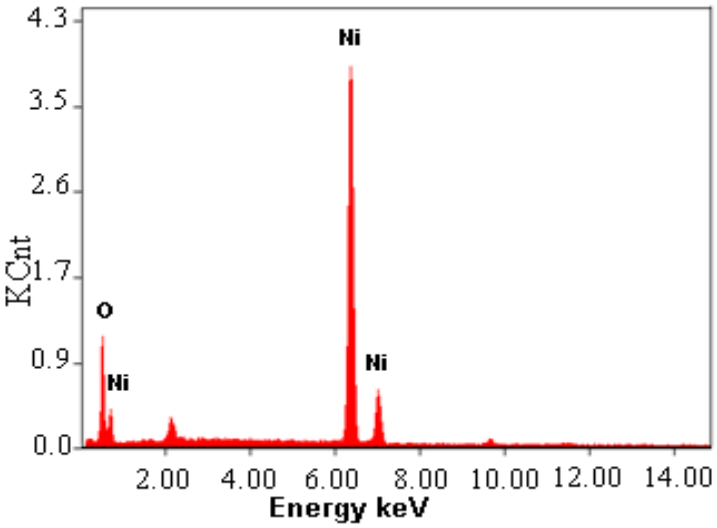

Fig. 4. EDX spectra of NiO nanoparticles.

3.5. TEM analysis

To provide further evidence for the formation of flakes –like nanoparticles morphology, TEM analysis was carried out and is shown in Fig. 5. From the image, we can conclude that the agglomerated nanoparticles morphology with below 50 nm in size. Selected area electron diffraction pattern (SAED) is shown in Fig.5, which implies that the prepared sample is well crystalline in nature.

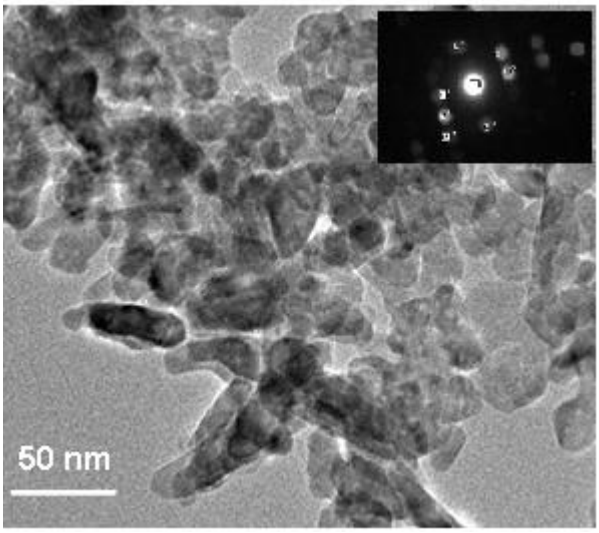

Fig. 5. TEM image and SAED patterns of NiO nanoparticles.

3.6. VSM measurements

Fig. 6 shows the VSM measurements of NiO nanoparticles in the applied filed range from -10 to +10 kOe at room temperature. From the VSM analysis, coercivity (H_c), remnant magnetization (M_r) and saturation magnetization (M_s) values has been obtained. M-H loop was confirmed that the superparamagnetic behavior. The M_s value of the sample is 6.35×10^{-4} emu/g, and M_r value is 1.352×10^{-4} emu/g respectively. The H_c value of the sample is 242.55 Oe.

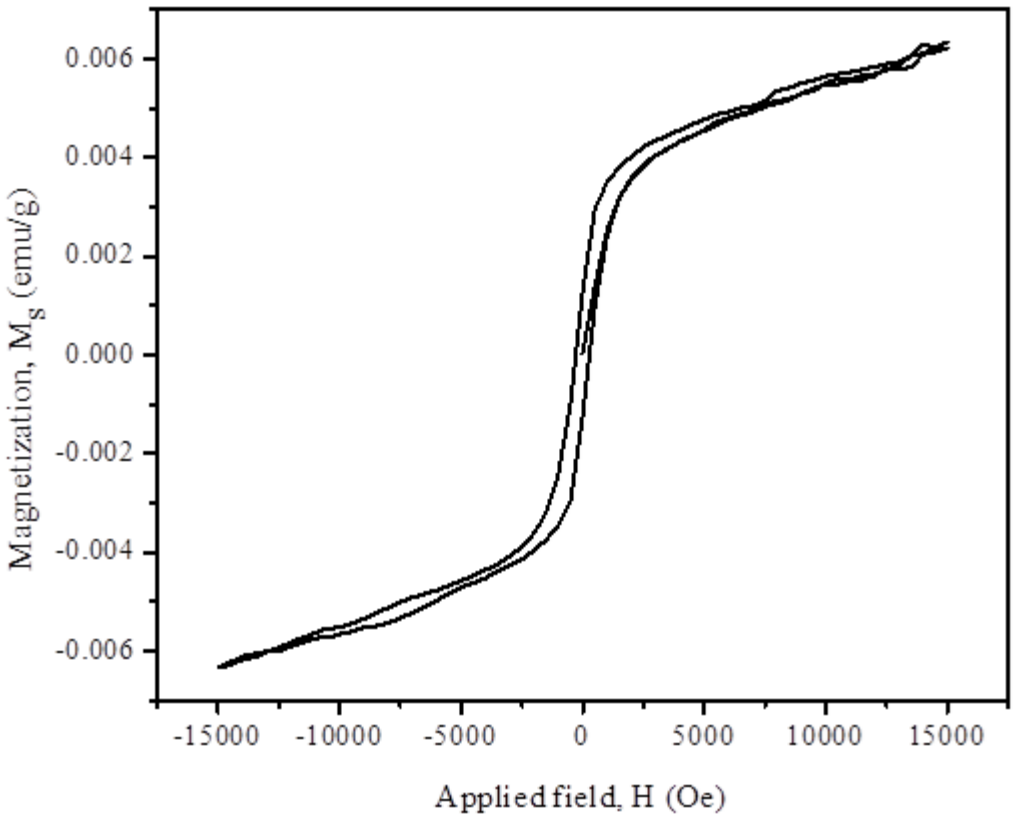

Fig. 6. VSM spectra of NiO nanoparticles.

4. Conclusions

Semiconductor NiO NPs have been successfully synthesized by a simple sol-gel combustion method using urea as the fuel. The formation of single pure phase was confirmed by powder XRD analysis. The average crystallite size was found to be 35.25 nm. The formation of flakes like nanoparticles morphology was confirmed by SEM and TEM analysis. Magnetic measurements revealed that the sample exhibited superparamagnetic behavior.

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