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Research Paper

Structural, optical and magnetic properties of cobalt oxide nanoparticles synthesized by simple combustion method

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Abstract

Cobalt oxide nanoparticles were synthesized by a microwave combustion method (MCM) for the first time, using urea as the fuel. The prepared samples were examined by XRD, HR-SEM, HR-TEM, UV-Visible spectra, and VSM. XRD analysis indicated well-crystallized cubic phase. HR-TEM images showed that the Co_3O_4 nanoparticles have sphere-like structure with the average particle size in the range of 25-50 nm. Optical properties of Co_3O_4 nanoparticles revealed the presence of two band gap (1.75 and 2.32 eV) values, which confirmed the purity and semiconducting properties. The VSM measurements revealed a small hysteresis loop at room temperature indicating a ferromagnetic nature.

Keywords: Cobalt oxide nanoparticles; Semiconducting properties; Ferromagnetic nature.

1. Introduction

Spinel Co_3O_4 nanomaterials have been widely considered as a promising type of multifunctional materials, due to their extensive applications in gas sensing, lithium-ion batteries, data storage, magnetic semiconductors, pigments, catalysis and electrochromic devices [1-7]. Many methods have been used to prepare Co_3O_4 nanostructures by physical and chemical

techniques such as sol–gel, co-precipitation, ball milling, and mechano-chemical methods [8-15], etc. Nevertheless, most of them were obtained through a wet-chemistry route. In fact, these processes often have disadvantages, such as requirement of toxic and costly reagents, expensive complex instruments, high-energy consuming and require rather long reaction times. Most of the methods are complex, low-yield and high-cost [16-20].

However, microwave combustion method has the priority over the above said conventional methods. It has attracted great attention, due to its simplest, easily scalable and energy-efficient technique for the fabrication of new catalytic materials. A template and complex apparatus are not needed. It also has potential advantages, including operational simplicity, high product purity, and no need for a solvent or special equipment [21-25].

In MCM technique, during the microwave combustion, the microwave energy interacts with the reactants at the molecular level, and the energy is transferred and converted in to heat. As a result of rapid heating it gives faster kinetics, homogeneity, higher yield, better reproducibility and energy saving [26-30]. Nano-sized particles prepared by using the MCM approach are expected to have a larger surface area, smaller particle size, and greater stability than those obtained by the other methods. Hence in the present study, we have prepared Co_3O_4 nanoparticles by MCM. The product was characterized by XRD, HR-SEM, HR-TEM, EDX, UV-Visible spectra, and VSM.

2. Experimental part

2.1 Preparation of Co_3O_4 nanoparticles

All the reagents used were analar grade obtained from Merck, India and were used without further purification. Stoichiometric amounts of cobalt nitrate ($Co(NO_3)_2.6H_2O$) and urea ($CO(NH_2)_2$) were dissolved separately in 10 ml of de-ionized water and poured into a silica crucible and stirred for 15 minutes to obtain homogeneous clear solution. This solution was placed in a domestic microwave-oven (2.45 GHz, 750 W) for 10 minutes. Initially, the solution boiled and underwent dehydration followed by decomposition with the evolution of gases. When the solution reached the point of spontaneous combustion, it was vaporized and instantly became a solid. The obtained solid was washed well with alcohol and dried in an air oven at 70°C for 1h.

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2.2. Characterizations

The structural characterization of Co_3O_4 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 of Co_3O_4 have been performed with a Jeol JSM6360 high resolution scanning electron microscope (HR-SEM). The transmission electron micrographs were carried out by Philips-TEM (CM20). The diffuse reflectance UV-Visible spectrum of Co_3O_4 was recorded using Cary100 UV-Visible spectrophotometer to estimate their energy band gap. The magnetic properties were investigated using vibrating sample magnetometer (VSM) at room temperature in an applied magnetic field sweeping from -10000 to +10000 Oe using a PMC MicroMag 3900 model vibrating sample magnetometer (VSM) equipped with 1 Tesla magnet.

3. Results and discussion

3.1. X-ray diffraction (XRD) analysis

Fig. 1 shows the XRD pattern of Co_3O_4 nanoparticles prepared by the microwave combustion (MCM). It can be clearly shown that the positions and relative intensity of the main diffraction peaks of the two samples were rather similar, and all the diffraction peaks could be perfectly indexed to the face-centered cubic Co_3O_4 phase [31]. The diffraction peaks could be assigned to (111), (220), (311), (222), (511), and (440) planes (JCPDS card No. 42-1467), respectively. No other phases such as CoO and Co_2O_3 can be identified. The very high peak intensity suggests that the material is highly crystalline. This indicates the complete transformation of precursor into the Co_3O_4 phase.

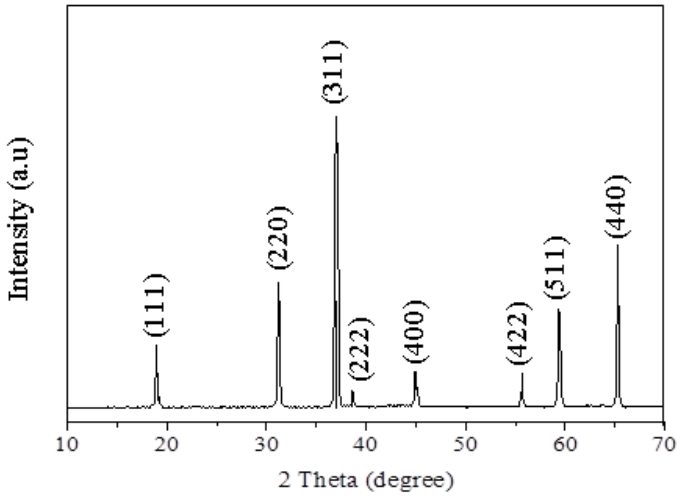

Figure 1. XRD patterns of Co_3O_4 nanoparticles

The average crystallite size of Co_3O_4 nanoparticles were calculated using Debye Scherrer 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 25.5 nm [32].

3.2 Scanning electron microscopy (SEM) studies

The surface morphology of the as-prepared Co_3O_4 nanoparticles was examined by HR-SEM method. Fig. 2 shows the HR-SEM images of the Co_3O_4 nanoparticles. A magnified HR-SEM image can be seen as a homogeneous nanoparticles consisting of agglomerated particles varying in size between 50 nm.

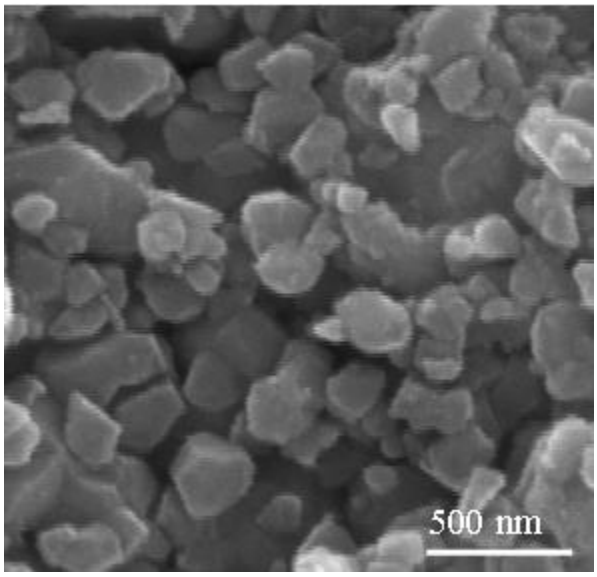

Figure 2. HR-SEM images of Co_3O_4 nanoparticles

3.3. Transmission electron microscopy (TEM) studies

The particle sizes of Co_3O_4 nanoparticles possess a narrow distribution, range from 20 to 25 nm (Fig. 3). The uniform Co_3O_4 nanoparticles have sphere like shapes with weak agglomeration. The average particle size determined by HR-TEM is very close to the average particle size calculated by the Debye-Scherer formula from the XRD pattern [33]. Selected area electron diffraction patterns (SAED) of the Co_3O_4 nanoparticles are shown in inset, which implies that the prepared Co_3O_4 nanoparticles are single crystalline in nature.

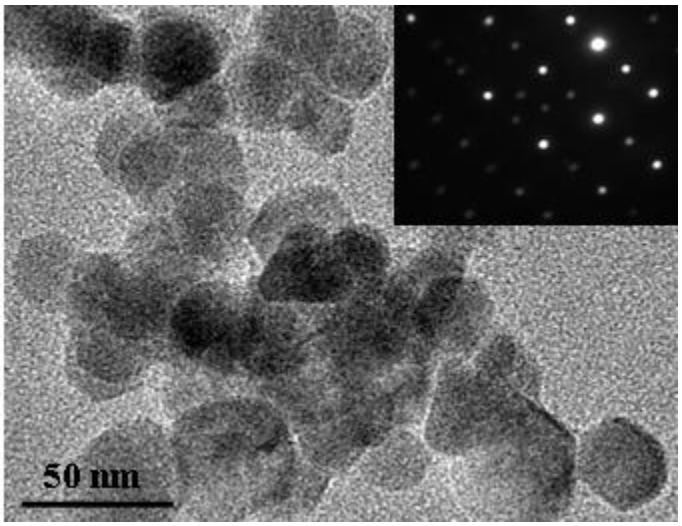

Fig. 3. TEM images of Co_3O_4 nanoparticles (inset of Fig, SAED pattern of Co_3O_4 nanoparticles)

3.4. Energy dispersive X-ray (EDX) analysis

Fig. 4 shows the EDX spectra of Co_3O_4 nanoparticles. The EDX results showed the presence of Co and O by the appearance of Co and O peaks without any other characteristic peaks. This observation further confirms that the final product is only Co_3O_4 nanoparticles. A small peak is appeared at 2.1 energy (KeV) for both samples, which indicates the presence of gold (Au) peak, that has been used as a coating, while preparing the sample for HR-SEM analysis for the better visibility of the surface morphology [34].

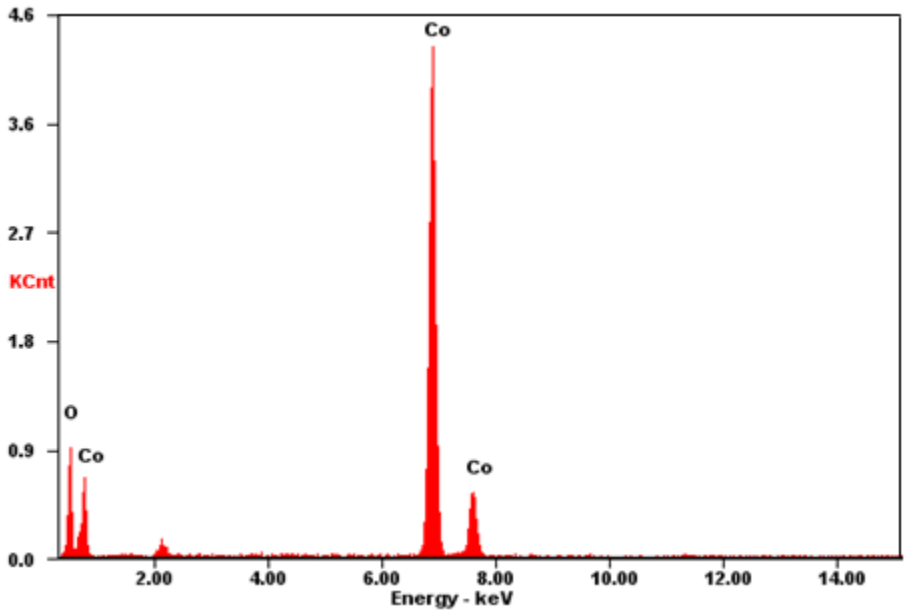

Figure 4. EDX spectra of Co_3O_4 nanoparticles

3.5 UV-Visible diffuse reflectance spectra

Spinel Co_3O_4 nanoparticles are semiconductor materials, which possesses direct transitions within the visible spectral region. The optical band gap (E_g) of the spinel Co_3O_4 nanoparticles can be calculated using the Kubelka-Munk (K-M) model [35] and the F(R) value is estimated from the following the formula given in Eq. (2):,

$$F(R) = (1-R)^2 /2R. \quad \text{---- (2)}$$

where F(R) is the Kubelka-Munk function, where R is the reflectance. The band gap can be estimated by extrapolating the linear region of the plot of $[F(R)h\upsilon]^2$ versus the photon energy as shown in Fig. 5, which can be attributed to O^{2-}–Co^{2+} charge transfer and O^{2-}–Co^{3+} charge transfer process [36]. Moreover, the appearance of two band gaps with the specific assigned values proves that the samples are pure and belong to the p-type semiconductor [37-40]. The increase in the band gap energies of the Co_3O_4 nanoparticles is an indicative of the quantum confinement effect arising from the tiny crystallites [41].

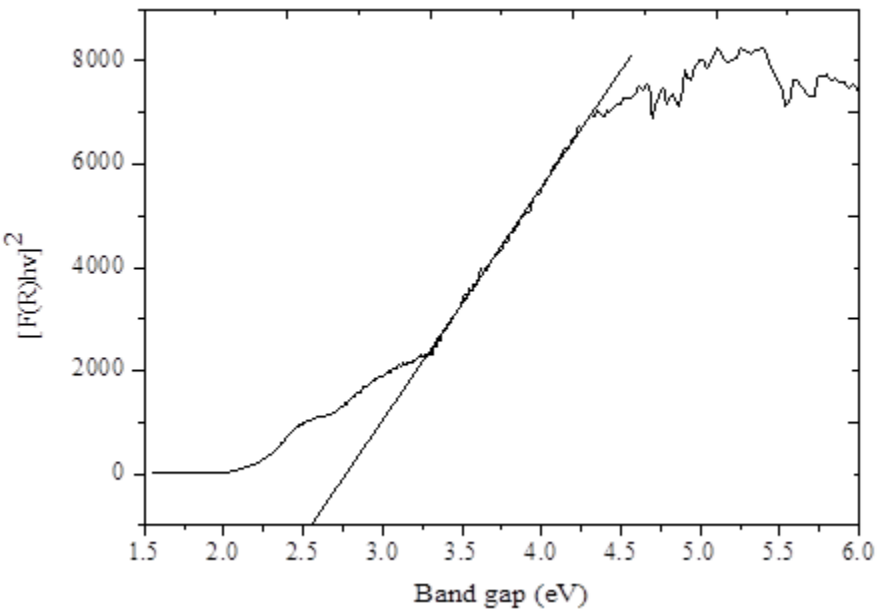

Figure 5. UV-Visible diffuse reflectance spectra of Co_3O_4 nanoparticles

3.6 Magnetic measurements (VSM)

Fig. 6 shows the M-H hysteresis of the Co_3O_4 nanoparticles. The shape of the hysteresis loop shows a characteristic weak ferromagnetic behavior, although bulk Co_3O_4 is anti-ferromagnetic [40]. The low H_c and M_r confirm that the Co_3O_4 nanoparticles have soft and weak ferromagnetic nature. But the bulk cobalt oxide (Co_3O_4) has a normal spinel structure with anti-ferromagnetic nature, due to the exchange between ions occupying tetrahedral and octahedral sites and has zero net magnetization, because of the complete compensation of sublattice magnetizations [41].

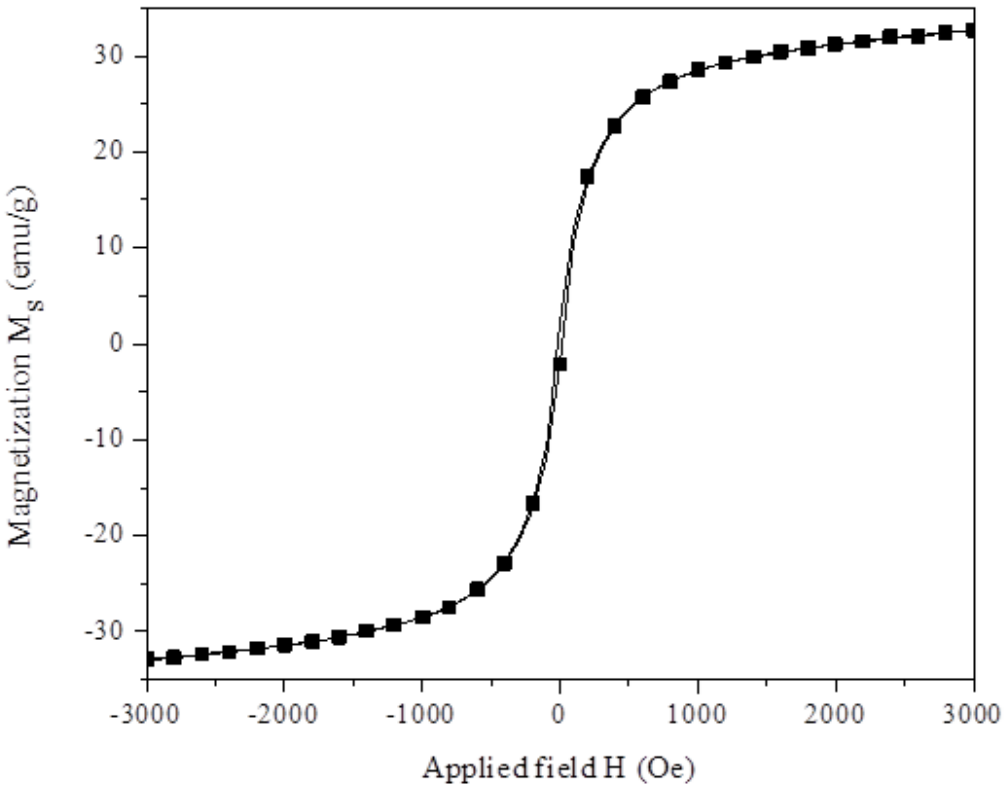

Figure 6. VSM measurements of Co_3O_4 nanoparticles

4. Conclusions

Nanocrystalline cobalt oxide was successfully prepared by eco-friendly microwave combustion method. Optical properties of the cobalt oxide nanoparticles revealed the presence of two band gap values confirmed the semiconducting properties. The low coercive fields and remanent magnetizations confirm that the Co_3O_4 nanoparticles have soft and weak ferromagnetic nature. Thus, this work provides a simple and convenient method for preparing the transition metal oxide having symmetrical structure. The synthetic route can be further used for the synthesis of other transition metal oxides.

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