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Green synthesis, characterization and optical properties of cobalt oxide nanoparticles by co-precipitation method

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Abstract

Cobalt oxide nanoparticles were prepared by eco-friendly Co-precipitation method using Cobalt Sulphate heptahydrate as precursor and sodium hydroxide as a stabilizing agent. The prepared nanoparticles were characterized by various techniques thermal analysis (TGA), X-ray diffraction (XRD), Scanning electron microscopy (SEM), Fourier transformer Infrared spectroscopy (FTIR), Energy dispersive X-ray analysis (EDXA) and UV-Visible spectroscopy. Optical properties showed an energy band gap i.e. direct and indirect band gap as 1.87 eV and 1.72 eV. The average particle size of cobalt oxide nanoparticles was found to be 25 nm from XRD.

Keywords: cobalt oxide nanoparticles, co-precipitation method, XRD, SEM, optical properties

1. Introduction

Nanostructured materials have wide range of applications in the various fields of studies especially in Optics, catalysts (water electrophoresis), electronics storage devices and biomedical science [1-4]. Cobalt oxide powders are used in applications such as lithium ion batteries, data storage, pigments and gas sensors [5-8] due to its physical and chemical properties [9]. Cobalt oxide is an important antiferromagnetic p-type semiconductor with high mechanical strength and direct band gap in the range 1.48-2.19 eV [10]. Co_3O_4 has a cubic spinel composed of cobalt in +2 and +3 valences. Co_3O_4 has a cubic spinel crystal structure in which the Co^{2+} ions occupy the tetrahedral sites and Co^{3+} ions the octahedral site [11]. Co-based nanomaterials like Co_3O_4, CoS, $CoFe_2O_4$, $NiCo_2O_4$ nanoparticles (NPs), Fe–Co bimetallic alloy NPs, show intrinsic catalytic activities and also used for the detection of H_2O_2, glucose, glutathione (GSH), and other biological materials [12]. There are different methods for cobalt oxide nanoparticles synthesis such as soft chemical method [13,14], hydrothermal [15,16], direct thermal decomposition [17,18], solvothermal synthesis [19], plasma spray pyrolysis [20], polymer based synthesis [21-23], microwave assisted synthesis [24,25] etc. but the above techniques require the complex equipment and long time for nanoparticles synthesis. The co-precipitation method is a simple process for the formation of controlled nanoparticles by using this method. In this study, a rapid and simple synthesis of Co_3O_4 NPs was reported and its structural and optical characterization by using UV Visible, XRD, FT-IR and SEM techniques.

2. Methods and Materials

2.1 Chemicals

All chemicals used in experiment were of analytical grade (AR). The chemicals used in the synthesis were Cobalt sulphate heptahydrate ($CoSO_4.7H_2O$, 98%) and sodium hydroxide (NaOH, 97%). All the solutions were prepared in double distilled water.

2.2 Synthesis of Co_3O_4 Nanoparticles

Cobalt oxide nanopowders were prepared by co-precipitation method. Cobalt sulphate heptahydrate (0.2M) and sodium hydroxide (0.1M) are mixed with continuous stirring at a constant temperature of 70 ^{0}C. While stirring, NaOH solution was added till the pH of solution become 12. The stirring was continued for 2 hours at a constant temperature of 70 ^{0}C. Reddish brown precipitates was formed, filtered and washed with acetone. Precipitates were dried over night at 100 ^{0}C. Than the precipitates was kept in muffle furnace at 500 ^{0}C for 2 hours. General scheme for the synthesis of cobalt oxide nanoparticles is shown Fig. 1.

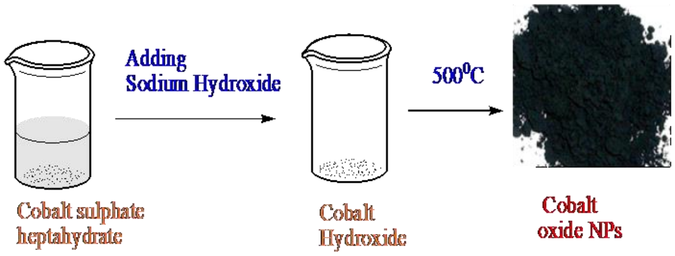

Fig 1: Scheme for the synthesis of cobalt oxide nanoparticles.

3. Characterization Techniques

The particle size, structure and morphology of as prepared nanoparticles are characterized by FTIR, SEM, UV-Visible and EDX spectroscopy. In FTIR, spectra were recorded on Nicolet 380 spectrometer in the range 400-4000 cm^{-1}. 10 mg of sample mixed with 100 mg of KBr in mortar and pestle and then applied a pressure of 7 tons into sample mixture and pressed into homogeneous pellets. Finally record the spectrum. Purity of the NPs was confirmed by powder XRD by Bruker (German) diffraction using CuKα radiation at a scanning rate of 5 ^{0}C/min. Structural morphology was examined by Hitachi S3700 SEM using an acceleration of 15 KV.

4. Results and Discussion

4.1 FTIR Spectra

Fig. 2 shows the FTIR spectra of an absorption peak at 571 cm^{-1} was assigned to Co-O stretching vibration mode in which Co^{+3} is octahedrally coordinated and peak at 658 cm^{-1} was assigned to bridging vibration in Co^{2+} is tetrahedrally coordinated [26, 27]. The peak at 1630 cm^{-1} and 3400 cm^{-1} are OH stretching and banding modes of water which is absorbed by the sample.

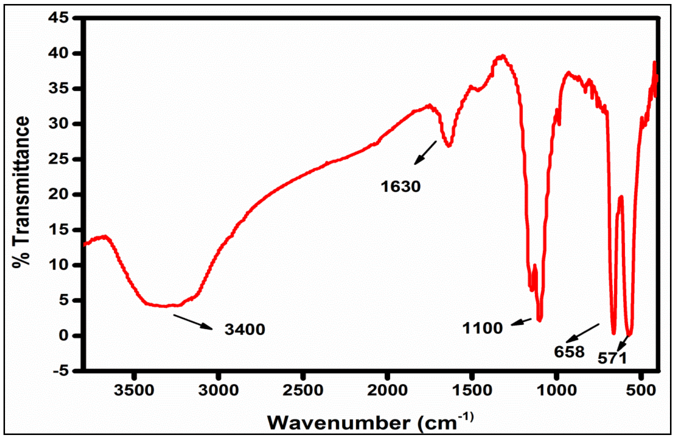

Fig 2: FTIR spectra of cobalt oxide nanoparticles.

4.2 XRD Analysis

The XRD pattern of fabricated cobalt oxide NPs is shown in Figure 3. The major peaks at 2θ values: 19.88 (111), 31.26 (220), 34.56 (311), 39.58 (222), 44.99 (400) corresponds to the simple cubic crystalline structure as established by JCPDS card file 073-1701. Average particle size of the Co_3O_4 nanoparticles was found to be 25 nm by using Debye Scherrer's equation (D = 0.9 k/ß Cos θ). In Scherrer equation 0.92 is a constant; k is the wavelength of the X rays and ß is the full width at half maximum of diffraction peak at θ is the diffraction angle.

4.3 UV-Visible Spectra

In Fig. 4, the optical properties of fabricated NPs show an absorbance peak at 460-580 nm by UV-Visible spectrophotometer. By using the Tauc's relation to calculate the direct and indirect band gap values are 1.87 eV and 1.72 eV respectively as shown in Fig. 5(a) and 5(b). The surface morphology of synthesized cobalt oxide NPs was investigated by SEM shown in Figure 6. The images of synthesized NPs are in higher to lower magnification i.e (5.00 µm and 3.00 µm).

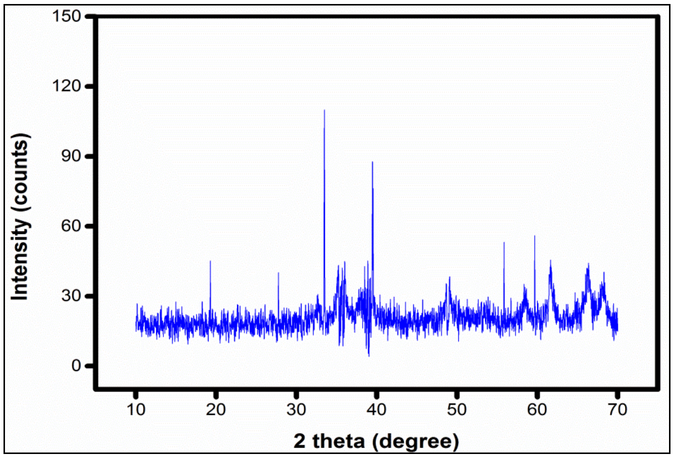

Fig 3: XRD spectra of cobalt oxide nanoparticles.

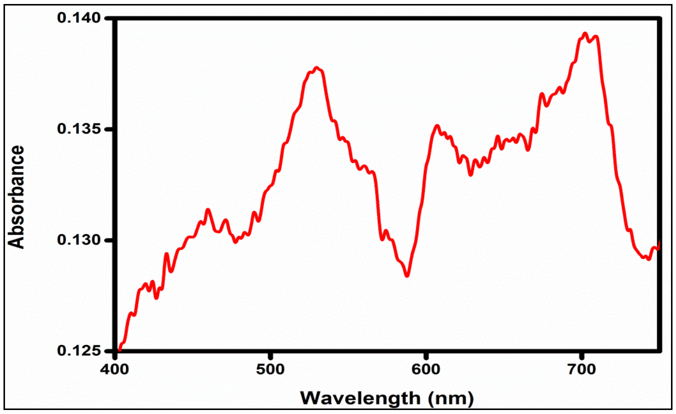

Fig 4: UV-Visible spectra of cobalt oxide nanoparticles.

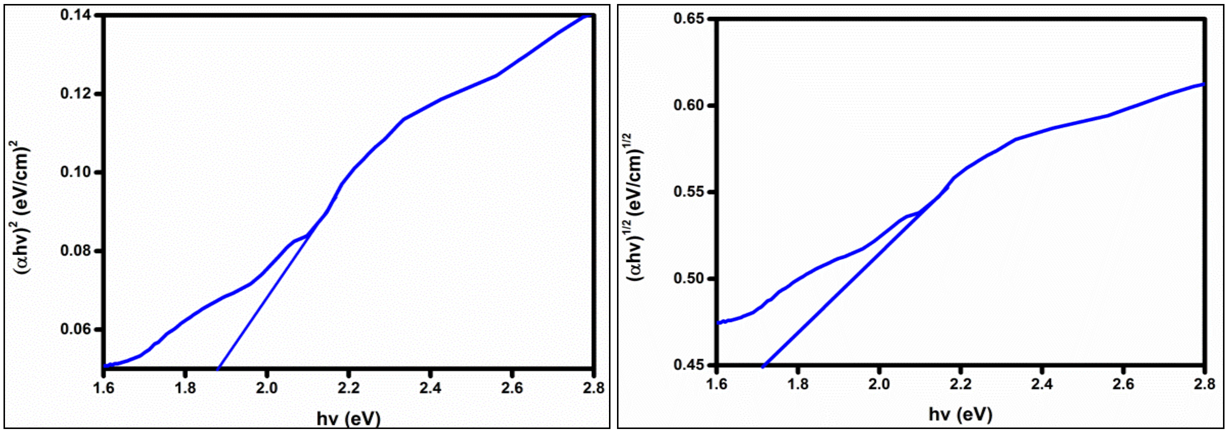

Fig 5: (a) and 5 (b): Direct and Indirect band gap spectra of cobalt oxide nanoparticles.

4.3 SEM Images

The cell structures of the samples were characterized with a Hitachi S3700 SEM (Scanning Electron Microscope) using an acceleration of 15 kV. Fig. 6 (a) and (b) show the scanning electron micrographs of the cobalt oxide nanoparticles.

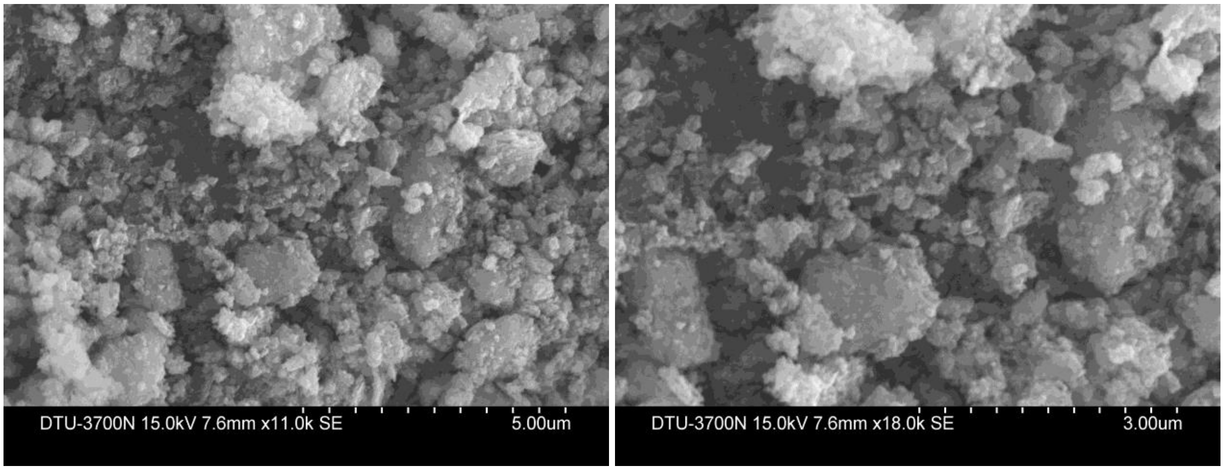

Fig 6: SEM Images of cobalt oxide nanoparticles.

In Fig. 7, the energy dispersive X-ray spectrometry (EDX) analysis was used to determine the elemental composition of as synthesized Co_3O_4 NPs. The EDX spectrum of Co_3O_4 contains cobalt and oxygen elements in atomic percentage are found to be 41.47% and 53.37% respectively and only 5.1% of Sulphur is present as an impurity in the sample.

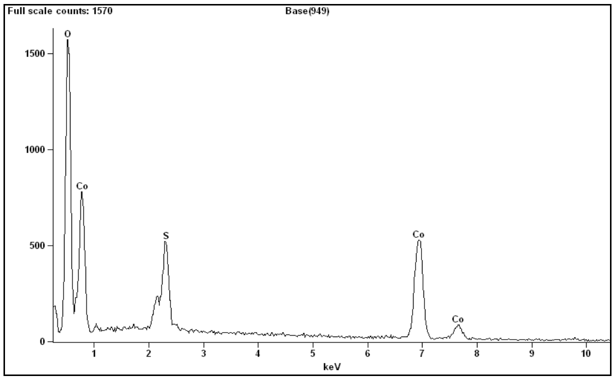

Fig 7: EDX Spectra of cobalt oxide nanoparticles.

Table 1: EDX Analysis of Cobalt Oxide –NPs

Element	Weight %	Atomic %
O (K)	24.66	53.37
S (K)	4.78	5.16
Co (K)	70.56	41.47
Totals	100.00	

Where “K” represent 1st inner shell

4.4 TGA Analysis

Fig. 8 (a) and (b) shows the spectra of TGA of cobalt oxide NPs that there are four Peaks on the derivative curve corresponding to the following temperatures: a maximum peak at around 85 ^{0}C which corresponds to water adsorption, a peak with minimum at 116 ^{0}C, followed by another at 333 ^{0}C and lastly a peak at 735 ^{0}C. Such effect can be described to the decomposition of Co_3O_4. TGA curve shows an evident decrease in the sample weight.

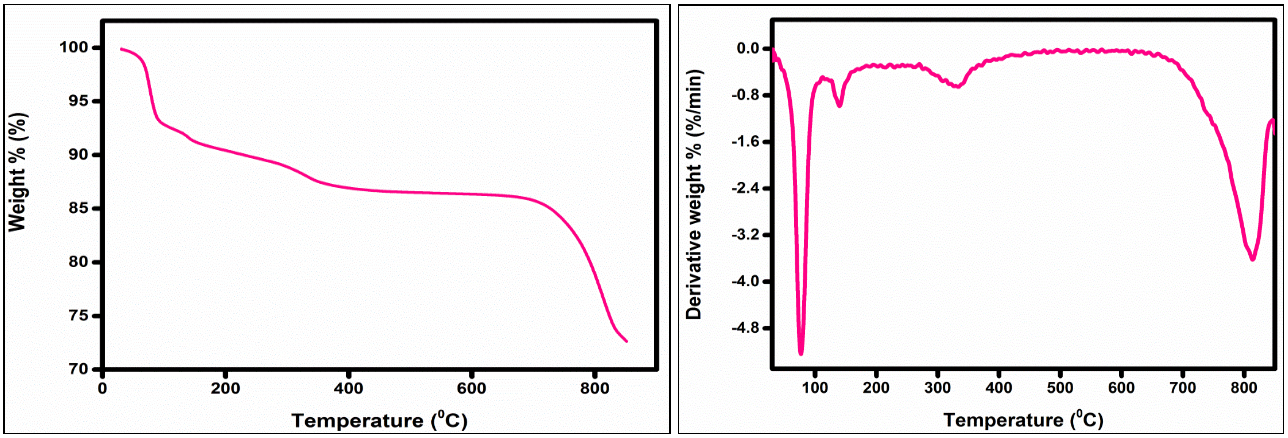

Fig 8: (a) and (b): TGA Curves of cobalt oxide nanoparticles.

5. Conclusion

The Cobalt oxide nanoparticles were fabricated by simple coprecipitation technique by using cobalt sulphate heptahydrate and characterised by advance techniques. XRD depicts the exact size of nanoparticle i.e 25 nm and optical properties shows an band gap around 1.87eV and p type semiconductor material.

6. References

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