Synthesis of Cobalt-Oxide Nanoparticle using Co-Precipitation Method

C. Nishitha, R. Jenisha, S. Sonia and S. J. Jenepha Mary*
Department of Physics, Holy Cross College (Autonomous), Nagercoil - 629004
Affiliated to Manonmaniam Sundaranar University, Tirunelveli - 627012
*Corresponding Author - Email: jenephaashok@gmail.com

ABSTRACT

In the present study, cobalt oxide (Co_3O_4) nanoparticles have been synthesized using cobalt nitrate as its source by precipitation approach. The structural properties of the synthesized Co_3O_4 nanoparticles were studied using X-ray diffraction (XRD). The XRD results confirm the crystalline nature of the nanoparticles. Further, scanning electron microscopy (SEM) was also performed to study the morphology of the synthesized Co_3O_4 nanoparticles. SEM micrograph shows that the Co_3O_4 nanoparticles are somewhat agglomerated sphere morphology.

Keywords: *Co-Precipitation process, Co_3O_4 nanoparticle, FESEM.*

1. Introduction

Syntheses and applications of metal and metal oxide nanoparticles have found paramount interest because of their unique properties due to their structure, dimension, morphology and size. Generally, transition metal oxides Co_3O_4 have been getting greater attention for electrochemical capacitor and sensor applications. Cobalt oxide (Co_3O_4), one of the transition metal groups metal oxide nanomaterials, is a good significant substance due to its exceptional characteristics and thermal permanence [1]. Characteristics as semiconducting, magnetic, optical, electrochemical, and electro-catalytic, cobalt oxide nanoparticles are widely used in heterogeneous catalysts, chemical sensors, photocatalytic hydrogen production, electrochromic devices, and energy storage systems [2,3]. Their enormous surface area and strong conductivity due to their nanoparticle size make them appropriate for catalysis. The cobalt oxide nanoparticles' high reversibility and ease of charge transfer during charging and discharging improve the device's performance [4]. Co-precipitation is a simple procedure that increases the size of nanoparticles while also controlling their development. The co-precipitation approach was used in this study to combine cobalt oxide nanoparticles for the purpose of electrochemicallydetecting blood cholesterol [5,6]. To the synthesis of cobalt oxide nanoparticles by simple chemical preparation method. The novelty of the work is the aqueous solution ammonium hydroxide is used for the synthesis process. Further the

structural, optical and morphological properties of the nanoparticles were analyzed using the characterization.

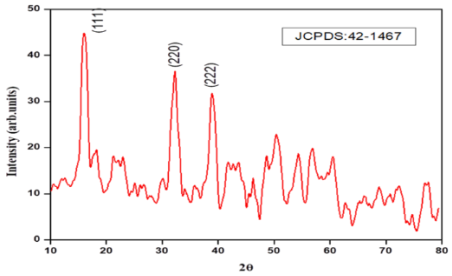
2. Experimental method

In the present work co-preparation process has been employed to synthesize cobalt oxide nanoparticles. 0.1M of cobalt chloride was added to 30ml of DI water. After that 30ml of ammonia hydroxide was mixed with 20ml of DI water. The ammonia solution was drop wise added to the transparent solution to rise the PH value 12. Then the solution was 30min of stirring and mixing. Then mixture was precipitate for 48hours. After that the precipitate was kept in oven at 90°C. The sample was finely ground into powder. Then, the sample was muffled at 500°C 2hr [7]. Finally, white powder is obtained.

3. Results and Discussion

3.1. XRD Analysis

The powder X-ray diffraction of the pure cobalt oxide nanoparticles is depicted in Fig.1. The intensity peaks observed at (111), (220) and (222) corresponding to 2ɵ =16.1778°, 2ɵ =31.8834° and 2ɵ =39.1018. The 2 values obtained from PXRD data of the as-prepared pure cobalt nanoparticles matched well with that of the JCPDS File No 42 - 1467. It is confirmed that the synthesized pure cobalt oxide nanoparticles exhibit a cubic structure. The sharp PXRD peaks clearly indicate the crystalline nature of the synthesized pure cobalt oxide Nano powder sample. The h k l and relative intensity values of pure cobalt oxide nanoparticles are presented.



XRD pattern of pure Co_3O_4

Position 2θ ($degree$)	FWHM (degree)	d-spacing (Å)	JCPDS file 2θ (degree)	(hkl)	Relative Intersity(%)
16.1778	0.3549	5.47892	5.47821	111	75.60
31.8834	0.4723	2.80688	2.80731	220	45.79
39.1018	0.4723	2.30375	2.31814	333	56.40

Table 1. Comparison of standard and observed d-spacing value

3.2. UV-Vis Absorbance Spectroscopy

To characterize the absorption properties of the synthesized samples, UV-Vis absorption spectrophotometer was used at a scan speed of 400 nm/min in the range of 200 am to 1000 nm. The transition of electrons between the valance band, conduction band, and inherent defect levels is responsible for the peaks seen in the absorption spectra. This is Co_3O_4 distinctive absorption peak. This is explained by Co_3O_4 inherent band-gap absorption. When visible particle size increases Absorption rises. The rise in oxygen vacancies could be the cause of this. Thereafter initializing the spectrometer with a glass and plate, the sample is attached for recording. Both absorption and transmittance. The pure Co_3O_4 nanoparticles' manufactured absorbance spectra are displayed in which has a maximum peak located at 297 nm.

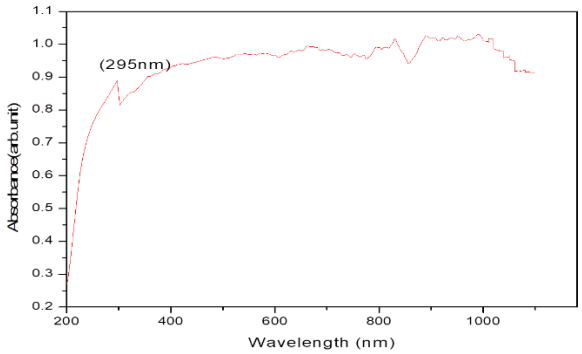


Fig.2 Optical absorbance spectra of pure CO_3O_4 nanoparticles

3.3. Fourier Transform Infared Spectroscopy

In practical applications, FTIR investigations aid in the analysis of potential bonding between adjacent structures. The spectroscopy that studies light with a longer wavelength and lower frequency than visible light, or the infrared section of the electromagnetic spectrum, is called Fourier transform infrared Spectroscopy. In comparison to their bulk counterpart, nanoparticles have a greater surface volume to ratio (also known as aspect ratio) as more molecules or atoms are placed on the nanoparticles surface.in order to rapidly determine whether the different modes included in CO_3O_4 nanoparticles are present or not. Using dry KBr as a standard reference, infrared spectra were acquired on an FTIR spectrometer within the 4000-500 cm-1 range. Below1500 cm-1, metal oxide typically produces absorption bands.

FTIR analysis confirms the characteristics of nanoparticles. The observed peak wave number of the frequency group are shown in the following table. The band 3316.60 cm^{-1}corresponds to the vibration of hydroxyl group. The band at 800 cm^{-1} corresponds to the presence of cobalt oxide nanoparticles [8].

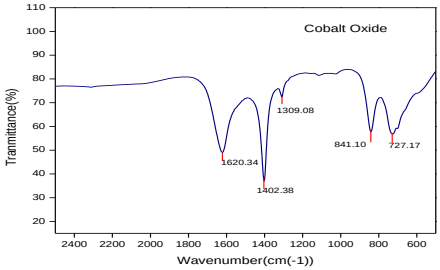


Fig 3. FTIR spectrum for as prepared Co_3O_4 nanoparticle

3.4. SEM Analysis

SEM Scanning electron microscopy (SEM) is widely used to study the size and morphology of the synthesized sample. SEM is based on a focused beam of electrons that scan the sample, which interacts with the atoms in the sample to provide three-dimensional surface topography. However, conventional SEM is performed under high vacuum and requires complex and extensive sample processing, including dehydration, fixation and metallization. SEM images of co-precipitation prepared samples are shown in figure. Nanostructures with various size and shape are obtained under different reaction parameters. It is observed that reaction temperature and duration influence morphology very much and hence the structural properties of nanoparticles. From the SEM images for synthesized at 0.1. CO_3O_4 nanoparticles are mostly in sphere shape [9].

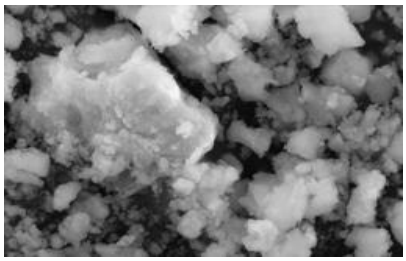


Fig.4 SEM images of ZnO particles synthesized using CO_3O_4at concentrations of 0.1 M

Conclusion

In the present work, nanoparticles of Cobalt oxide were synthesized by a simple Co-precipitation method. The precursors used in the synthesis process were Cobalt chloride and Ammonium hydroxide. The preparation process is faster, cheaper and cost effective. The following conclusions are made from the result of this work. From the XRD characterization, it is observed that the both cobalt oxide nanoparticles belong to cubic structure. Phase identification was carried out using XRD analysis. The average particles size was calculated from the W-H plot and the Scherer's formula. The peaks that were acquired line up with standard file JCPDS file number: 42-1467. The average particle grain size, D = 0.18888 X

10^{-9} nm, and the dislocation density, δ = 0.0002957 X 10^{15} lines/m2, are determined. Along the (111) plane, every particle has a preferred orientation. The absorbance of the as-prepared sample was examined using UV characterization, and it was found that the optical band GAB had an absorbance of 0.92 (arbitrary unit) with a maximum peak at 356 nm.

The optical properties of the prepared nanoparticles were analyzedusing UV-Vis spectrophotometer. All the three prepared nanoparticle shows lower absorbance percentage which confirms that the nanoparticles have higher transmittance. The calculated band gap proves that cobalt oxide nanoparticles are semiconductor. The fact that the nanoparticlesare agglomerated to form spherical-shaped particles in the SEM analysis.

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