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Hexagonal Cobalt Oxide Nanoparticles: A Cost-Effective Route to High-Performance Functional Materials

Jyoti Mayekar
Department of Physics, Jai Hind college, India

Abstract : *This study presents the synthesis of hexagonal cobalt oxide (Co_3O_4) nanoparticles using a simple two-step process involving chemical co-precipitation and thermal decomposition. The method provides a cost-effective and eco-friendly alternative to conventional synthesis techniques by avoiding toxic solvents and complex equipment. The resulting nanoparticles were thoroughly characterized using X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDAX), and scanning electron microscopy (SEM), confirming their high purity, single-crystalline structure, and nanoscale dimensions (~30 nm). Due to their unique structural and electronic properties, Co_3O_4 nanoparticles exhibit significant potential for a wide range of applications, including energy storage, catalysis, and sensor technologies. Their favorable bandgap and high surface area contribute to enhanced electrochemical performance, making them promising candidates for next-generation supercapacitors and functional materials. This work provides a simple yet effective synthesis route for cobalt oxide nanomaterials, paving the way for further research into their technological and industrial applications.*

Keywords - Nanotechnology, *Cobalt oxide nanoparticles, metal oxide nanoparticles nanotechnology, scanning electron microscopy X ray diffraction*

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I. INTRODUCTION

Metal oxides and hydroxides play a crucial role in electroactive materials, with cobalt oxide (Co_3O_4) being a prominent transition metal oxide due to its excellent electrochemical properties. It has garnered significant attention as a pseudocapacitive material for supercapacitor applications, offering a promising alternative to RuO_2 owing to its high theoretical capacity, low cost, wide availability, and environmentally friendly nature. Over the past decade, substantial progress has been made in synthesizing nanoscale Co_3O_4 with controlled size, structure, and morphology to enhance its capacitive performance. Various morphologies of Co_3O_4, including nanowires, nanosheets, nanoflowers, nanoparticles, nanorods, urchin-like structures, and hollow nanostructures, have been successfully synthesized and utilized as key components for high-performance supercapacitors [1].

Co_3O_4 is a p-type semiconductor with a cubic spinel crystal structure, where Co^{2+} ions occupy tetrahedral sites and Co^{3+} ions are present in octahedral sites. The diamagnetic nature of Co^{3+} in the octahedral crystal field and the antiferromagnetic sublattice formed by Co^{2+} contribute to its unique magnetic and electronic properties. Cobalt spinel compounds have also demonstrated their efficacy as catalysts in various heterogeneous chemical processes. Due to its exceptional properties, Co_3O_4 nanoparticles are widely used in electronic devices, gas sensors, electrochromic devices, lithium-ion batteries, and high-temperature solar selective absorbers [2].

While many metal oxides, including ZnO, Al_2O_3, TiO_2, NiO, CeO_2, CuO, and MgO, exhibit cytotoxicity, oxidative stress induction, DNA damage, and inflammation risks, Co_3O_4 presents a comparatively safer alternative. For example, ZnO nanoparticles have shown significant toxicity to human pulmonary cells, whereas TiO_2 nanoparticles are known for their relatively benign environmental impact. The abundance, cost-effectiveness, and chemical stability of cobalt oxide make it an attractive material for large-scale production and industrial applications. Additionally, Co_3O_4 features a favorable bandgap (1.48–2.19 eV), making it highly suitable for applications in capacitors and other energy storage devices [3].

Nanostructured metal oxides, including Co_3O_4, have been extensively studied for their unique optical, magnetic, catalytic, and electrochemical properties, which enhance their usability in advanced material applications. The reduction of Co_3O_4 to nanoscale dimensions further improves its performance in gas-sensing, catalysis, supercapacitors, and solid-state sensor applications. As a result, researchers have actively explored various synthesis techniques to optimize the properties of Co_3O_4 nanostructures for practical applications [4-8].

Cobalt-based nanoparticles are considered among the most promising materials for technological advancements in information storage devices, magnetic fluids, and catalysts. Cobalt itself is a well-known

ferromagnetic material, commonly used as an alloying element in permanent magnets. It exists in two primary crystalline phases: hexagonal close-packed (HCP), which is stable at room temperature, and face-centered cubic (FCC), which is stable at temperatures above 450°C. Additionally, cobalt oxide nanoparticles find applications in fields such as separation technology, biomedicine, and catalysis. To achieve optimal performance, nanoparticles must be uniform in size, shape, and composition, necessitating thorough characterization [9]. Moreover, recent studies have explored the potential of cobalt oxide nanoparticles in anticancer treatments, further broadening their biomedical applications [10].In our earlier work zinc oxide, copper oxide and magnesium oxide nanoparticles have been studied[11,12,13]. In future comparative study of these metal oxide nanoparticles can be done.

II. EXPERIMENTAL

All chemicals used in this study were of analytical grade and used without further purification. Cobalt oxide nanoparticles were synthesized using cobalt nitrate as precursor and sodium hydroxide as a reducing agent. In order to synthesize stable nanoparticles and avoid agglomeration starch is used as a capping agent.2.910 gms of cobalt nitrate along with 0.1 gms of starch is dissolved in 100 ml of double distilled water. Stir the solution using magnetic stirring and heat the solution till the temperature reaches 60^0C. Once the desired temperature is achieved, stir the solution for 1 hr. Keep it overnight. Centrifuge and wash the precipitate with distilled water for five times. Dry the precipitate at 100^0C for 2 hrs. Brown- colored powder is obtained. Heat the powder in a muffle furnace for 6 hrs at 400^0C. Black-colored powder is obtained. Using the present method, Co_3O_4 nanoparticles can be produced without the need of expensive organic solvents and complicated equipments. The addition of NaOH is an alkali which is used to maintain the pH of the solution. The OH^- ions have a major role behind the formation of nucleation sites which determine the shape of the crystal. The OH^- ions present in the solution will affect surface energies due to steric effect. The growth of the other facets will be inhibited and this will lead to the formation of particles in different shape such as rods of one dimension.

III. RESULTS AND DISCUSSIONS

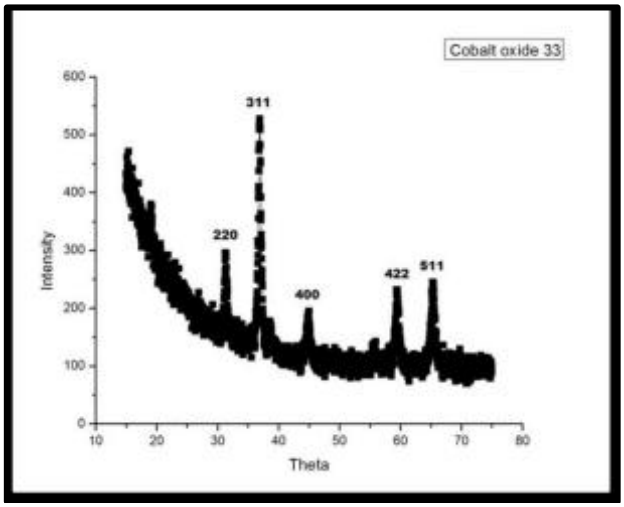

Figure 1 X- ray diffraction spectroscopic image of synthesized cobalt oxide nanoparticles

The diameter of the nanoparticles calculated by the Scherer formula is 30 nm. No other peaks for impurities were detected.

The phase formation and crystallographic state of cobalt oxide NPs were determined by XRD with an ExpertPro (Phillips) Xray diffractometer using Co Kα radiation. Samples were scanned from 20° to 80° of 2θ increment of 0.04° with 2 s counting time.

The XRD pattern proves the Co_3O_4 stable single phase formed as cubic normal spinel structure and its lattice parameters a=b= c=8.072Å and space group of Fd3m, which are consistent with those reported (JCPDS card No 76-1802). Almost all of peaks intensities of (111), (220), (311), (222), (400), (422), (511), (440), can be perfectly indexed to a pure cubic phase Since the peak from the most intense (220) plane diffraction was selected to calculate the average crystallite size of Co_3O_4.There were no characteristic peaks of impurities.The crystallite size was calculated using Debye Scherer formula

$$D = \frac{0.9\lambda}{\beta cos\theta}$$

Where λ is the wavelength of Kα radiation, β is full width half maximum(FWHM) in radians and θ is angle of incident radiation in radians.

The lattice parameter a can be calculated using the relation

$$d^2 = \frac{a^2}{h^2 + k^2 + l^2}$$

Where d is the interplanar distance and h, k and l are miller indices.

The density ρ of the samples were calculated from the formula

$\varrho = \frac{nM}{NV}$ (gm/cm^3)

Here n represents the no. of atoms in the unit cell, M denotes the molecular weight of the sample, N is the Avogadro number and V is the volume of the unit cell, M denotes the molecular weight of the sample, N is the Avogadro number and V is the volume of the unit cell.

The specific area of the sample calculated using the formula

$Sa = \frac{6}{\varrho\ X\ D}$cm^2/g

Where Sa is the surface specific area, ρ is the density of the samples (gm/cm^3) and D is the crystallite size of the sample (nm).

The microstrain of the samples was calculated using the relation

$$\varepsilon = \frac{\beta cos\theta}{4} lin^{-2}m^{-4}$$

Parameter	**Value**
Lattice parameter a A^0	8.072
Volume V $(A^0)^3$	525.95
Density (ρ) (gm/cm^3)	6.11
Crystallite size (D) (nm)	30
micro strain (ε) X10^{-4} (lin^{-2} m^{-4})	3.25x 10^{-3}
Specific surface area Sa (cm^2 /g) X 10^6	3.27 x 10^{-6}

Table 1: Structural parameter values of synthesized cobalt oxide nanoparticles

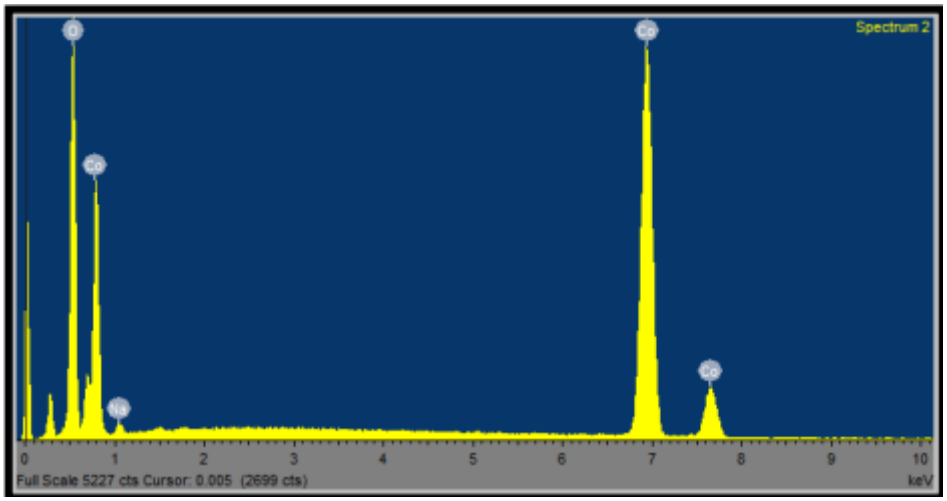

Figure 2: Energy dispersive spectroscopic graph of synthesized cobalt oxide nanoparticles

Spectrum	**Co (at wt %)**	**O (at wt %)**
Spectrum 1	45.68	54.32
Spectrum 2	39.11	60.88
Mean	4.40	57.6
Std. deviation	4.65	4.65
Max.	45.68	60.88
Min.	39.11	54.32

Table 2: Elemental composition of synthesized cobalt oxide nanoparticles

Chemical purity and stoichiometry of the Co_3O_4 was tested by EDX. The strong peaks related to Co and O is found in the spectrum Fig. 4. EDX spectrometry for nanoparticles shown oxygen that confirm sample was Co_3O_4.

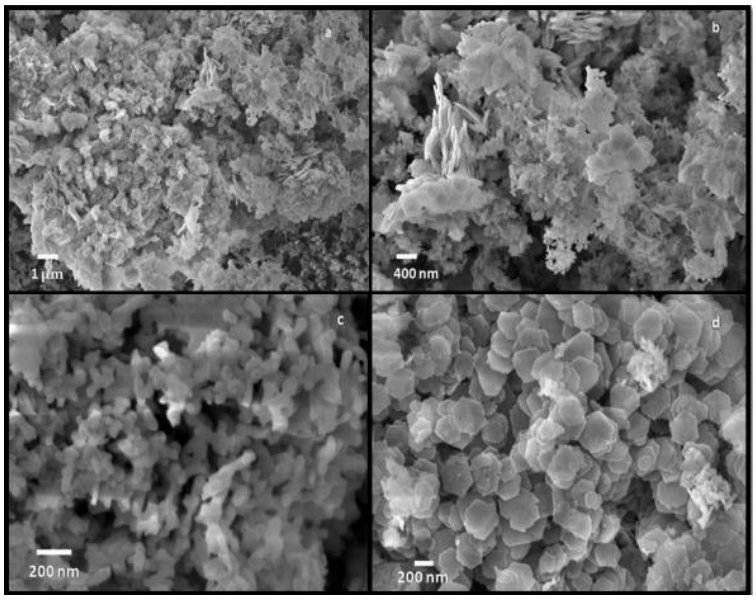

Figure 3 : Scanning electron microscopic images of cobalt oxide nanoparticles at different magnification

The morphology of the Co_3O_4 nanoparticles is examined by SEM. The nanoparticles are found to have hexagonal , morphology. The particle size is of the order of 100-200 nm.

IV. CONCLUSION

In this study, cobalt oxide (Co_3O_4) nanoparticles were successfully synthesized using a simple and eco-friendly two-step process involving chemical co-precipitation and thermal decomposition. The approach eliminates the need for toxic solvents or complex equipment, making it a cost-effective method for large-scale production. Characterization using XRD, EDAX, and SEM confirmed the formation of pure, single-crystalline Co_3O_4 nanoparticles with a cubic spinel structure and an average crystallite size of 30 nm.The synthesized nanoparticles exhibit desirable properties, including high chemical stability, structural integrity, and a favorable bandgap, making them well-suited for various applications. Their potential spans across fields such as energy storage (supercapacitors and batteries), catalysis, gas sensing, and biomedical applications, including anticancer treatments. Future research could focus on optimizing synthesis parameters to achieve better control over particle size and morphology. Additionally, further exploration of their performance in real-world applications, such as advanced energy storage systems and biomedical uses, could unlock new possibilities for cobalt oxide-based nanomaterials.

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