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HYDROTHERMAL SYNTHESIS AND CHARACTERIZATION OF COBALT (II) OXIDE (CoO) NANOPARTICLES

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ABSTRACT

This work presents a straightforward and effective hydrothermal method for synthesizing cobalt (II) oxide (CoO) nanoparticles with controlled physicochemical properties. The obtained nanoparticles were fully characterized by UV-Vis spectroscopy, FTIR, XRD, photoluminescence (PL), Raman spectroscopy, and high-resolution TEM-EDAX studies. XRD investigation showed the presence of a face-centered cubic crystal structure. FTIR spectra confirmed phase purity and identified key functional groups. The UV-Vis spectrum showed distinct absorption bands, whereas the PL spectrum indicated a significant emission peak at 610 nm, indicating surface flaws or electronic transitions. TEM pictures revealed polycrystalline nanoparticles with homogeneous shape, which was corroborated by SAED patterns. Raman spectra indicated that the vibrational modes correspond to the CoO phase. The study demonstrates that hydrothermal synthesis is a sustainable and economical method. For producing high-quality CoO nanoparticles, which show promise for applications in optoelectronics, sensing, and catalysis.

Keywords: Cobalt Oxide Nanoparticles, Hydrothermal Synthesis, Optical Properties, Photoluminescence, Raman Spectroscopy, TEM, Structural Characterization.

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INTRODUCTION

Nanomaterials are transforming the landscape of sustainable energy, electronics, and environmental monitoring. Their exceptional properties, driven by their ultra-small dimensions, offer unparalleled opportunities for developing smart and sustainable technological solutions.[1] Because of their primarily high surface-to-volume ratios, transition group metal nanoparticles and their oxides are frequently used as catalysts in petrochemical and industrial processes as well as energy research.[2] Metal oxides possess an array of chemical and physical properties due to their ability to take on a wide range of structural geometries and an electronic structure that can display features of an insulator, semiconductor, or metal.[3] Therefore, metal oxides serve as key functional materials for chemical and biological sensing and signal transmission.[4] Furthermore, they are outstanding choices for optoelectronic and electrical applications due to their distinct and adjustable physical characteristics. Chemical or physical procedures can be used to create nanomaterials. Physical methods include methods such as spray pyrolysis and physical vapor deposition (PVD).[5,6]However, chemical procedures include sol-gel processes, chemical vapor deposition (CVD), hydrothermal and non-aqueous synthesis, and micro emulsion techniques.[7,8,9,10, 11]It's significant to consider how variations in morphology, or shape or structure, can have considerable effects on a material's qualities even when those components have the same chemical structure. Controlling the morphology during chemical synthesis to customize material performance has become more and more popular as a result.[12] As a result, several morphologies and architectural styles of nanomaterials have been produced.[13]Despite previous findings on CoO nanoparticles, few investigations have focused on fine-tuning the hydrothermal synthesis process to maximize crystallinity, shape, and defect-related optical characteristics. This study fills that gap by using a simple, environmentally friendly hydrothermal method and performing extensive structural and optical analyses.[14] The magnetic p-type semiconductor cobalt (II, III) oxide (CoO) is a significant component of the conventional spinel structure, with an array of oxide ions arranged in a cubic tight packing.[15] In this reaction study, we demonstrate the process of

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manufacture, the characterization of the produced nano oxides using different techniques, and some intriguing findings regarding the CoO nanoparticles.[16] Because it is easy to perform, hydrothermal synthesis is often the method of choice for synthesizing metal oxide nanostructures. A Teflon-lined autoclave containing the required quantity of powdered reagents and water is heated without stirring, gradually reaching moderate to high temperatures and pressures over the desired time. Furthermore, by adjusting the precursor ratios in the hydrothermal synthesis, the impurity problem can be solved, and the ideal reaction conditions can be predicted using electrolyte thermodynamics.[17]

EXPERIMENTAL

Materials and Methods

Every chemical utilized was of analytical reagent quality and was used without further purification. The synthesis was carried out using deionized water. Cobalt (II) acetate tetrahydrate ($C_4H_6CoO_4.4H_2O$) and sodium hydroxide (NaOH) were employed for the preparation of CoO. Typically, the process begins with cobalt (II) acetate tetrahydrate, hexamethylenetetramine (HMTA), and sodium hydroxide dissolved in surfactant-containing solvents.

Experimental Procedure and Synthesis of Cobalt Oxide Nanoparticles

Initially, Cobalt (II) acetate tetrahydrate (0.1 M, 0.249 g in 40 mL deionized water) was magnetically stirred for 30 minutes. Then, 0.1 M hexamethylenetetramine (HMTA) was added and agitated for 15 minutes. Sodium hydroxide (0.5 M) was then added dropwise while swirling continuously to create a homogeneous solution. The mixture prepared was loaded into a 100 mL Teflon-lined stainless-steel autoclave, tightly closed, and heated at 180 °C for 8 hours. Subsequently, allowed to cool to room temperature, the precipitate formed in the resulting mixture was separated, washed thoroughly with deionized water and ethanol, dried at 80 °C, and then annealed at 600 °C for 1 hour.

RESULTS AND DISCUSSION

The structural analysis of the synthesized Cobalt (II) oxide (CoO) nanoparticles was performed using powder X-ray diffraction (XRD) on an ARL EQUINOX 3000, Cu Kα radiation source in a diffractometer (λ = 1.54 Å). CoO nanoparticles' optical characteristics were examined using a UV–Visible spectrophotometer (UV-2101, Shimadzu) with wavelengths ranging from 200 to 800 nm. FTIR (Fourier transform infrared) spectra were obtained using an ALPHA-T spectrometer equipped with an ATR accessory (Bruker, Germany), with samples prepared using KBr pellets. Fluorescence measurements were conducted using an F-7100 FL spectrophotometer. Raman spectroscopy was carried out with a Renishaw inVia confocal Raman microscope (United Kingdom). High-resolution scanning transmission electron microscopy (HRSTEM) was used to analyze the CoO nanoparticles' shape and elemental distribution. The images were recorded on a TALOS F200S G2 microscope with an accelerating voltage of 200 kV. TEM characterization was done by preparing samples by dropping a drop of diluted ethanol suspension onto copper grids and drying. Elemental mapping of the distribution of CoO was also further analyzed using energy-dispersed X-ray spectroscopy (EDS) mapping.

XRD Analysis

The synthesized cobalt oxide nanoparticles XRD study (Figure 1) shows clear peaks in the 2-theta at 18.89°, 31.38°, 36.84°, 38.24°, 44.93°, 55.64°, 59.52°, and 65.22°. The JCPDS file number 65-3103, which is associated with a cobalt oxide phase, matched these peaks. A face-centered cubic (FCC) lattice structure and the cubic system are both compatible with the observed diffraction pattern.[18] The effective synthesis of cobalt oxide nanoparticles with the anticipated crystalline phase and structure is confirmed by the conformity of the experimental peaks with the JCPDS reference file.

UV- Vis Analysis

As seen in Fig.-2, the UV–Vis absorption spectrum of CoO nanoparticles shows two distinct peaks at about 266 nm and 394 nm. These absorption peaks might be associated with cobalt oxide electronic transitions. The size of the nanoscale particles and the existence of surface states or lattice defects most likely have an impact on the peak broadening.[19]

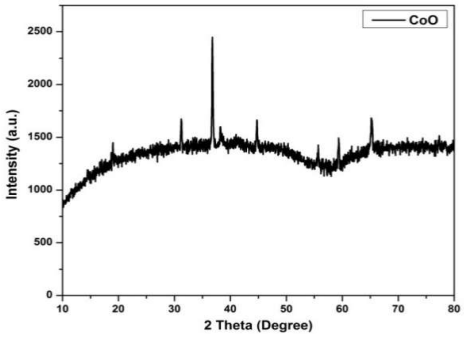

Fig.-1: XRD Patterns of CoO Nanoparticles

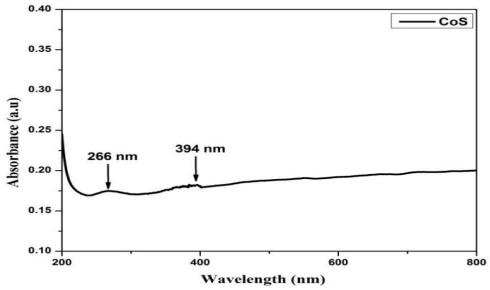

Fig.-2: UV-Vis Absorption Spectrum of CoO Nanoparticles

FTIR Analysis

From Figure 3, The OH^- stretching vibration at 3422 cm^{-1} and the weak asymmetric band at 1627 cm^{-1} confirm the existence of OH^- groups, which are likely due to water adsorption during sample preparation. Intense M–O stretching at 664.69 cm−1 and bending at 568.74 cm^{-1} confirm the face-centered cubic structure of the nanoparticles, phase purity, and monodispersity. The Co–O bending and stretching vibrations are at 440 cm−1 and 585 cm^{-1}. The bands at 1106 cm^{-1} and 1272 cm^{-1} are due to the stretching vibrations of C–O, which confirm the existence of carbonate groups or trace organic compounds. Peaks present at 1413 cm^{-1} and 2026 cm^{-1} exhibit C–H bending and stretching, whereas C–H stretching is responsible for peaks at 2855 cm^{-1} and 2924 cm^{-1}, indicating the existence of hydrocarbons or organic pollutants.[20,21]

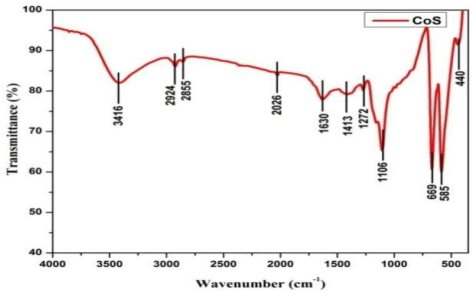

Fig.-3: FT-IR Spectrum of CoO Nanoparticles

PL Analysis

Figure-4 shows a single, strong emission peak centered at 610 nm in the photoluminescence (PL) spectrum captured at an excitation wavelength of 300 nm. Defect-related radiative recombination processes, which are frequently linked to surface states or oxygen vacancies seen in cobalt oxide nanoparticles, could be the cause of this emission.[22]

TEM Measurements

TEM analysis revealed polycrystalline CoO nanoparticles with irregular shapes and a narrow size distribution, likely resulting from surface interactions. Figure-5 shows the HR-TEM images, which reveal

the crystalline structure of the particles, and the corresponding SAED pattern shows well-defined diffraction rings, confirming their polycrystalline characteristics. Additionally, energy-dispersive X-ray spectroscopy (EDS) mapping demonstrates a uniform elemental distribution. The combined TEM, HR-TEM, SAED, and EDS results provide strong evidence of the structural consistency and purity of the synthesized CoO nanoparticles.[23]

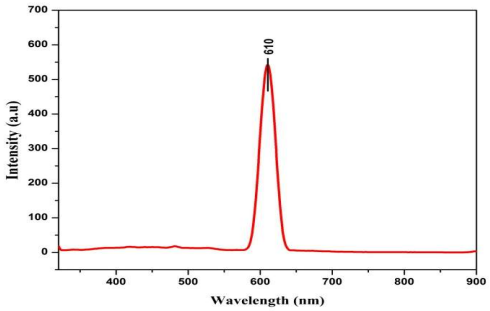

Fig.-4: PL Spectrum of CoO Nanoparticles

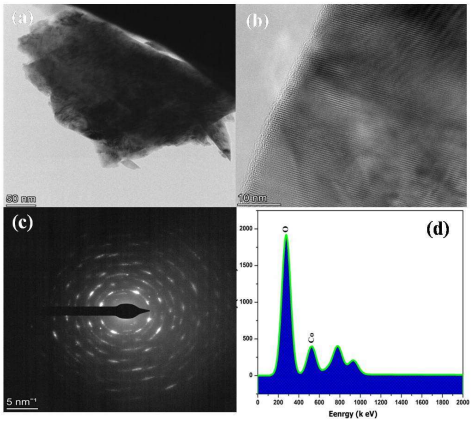

Fig.-5: (a) TEM, (b) HR-TEM Images, (c) SAED Pattern, and (d) EDS of Cobalt Oxide Nanoparticles

Raman Analysis

The Raman spectrum of CoO nanoparticles with peaks at 190, 469, 513, and 674 cm^{-1} reflects the vibrational modes typical of cobalt oxide. The CoO lattice vibrations are responsible for the peak at 190 cm^{-1}, whereas the optical phonon modes are linked to the peaks at 469 and 513 cm^{-1}. Figure-6 shows that the presence of cobalt oxide with its distinctive characteristics is confirmed by the peak at 674 cm^{-1}, which is linked to extra vibrational modes of the CoO structure.[24]

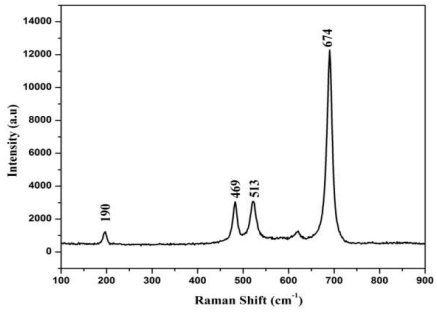

Fig.-6: Raman Spectrum of CoO Nanoparticles

CONCLUSION

The hydrothermal production of cobalt (II) oxide (CoO) nanoparticles with distinct structural and optical characteristics is successfully demonstrated in this work. A face-centered cubic crystal structure was confirmed by XRD analysis, and the presence of Co–O bonding and CoO-specific vibrational modes was

established by FTIR and Raman spectroscopy. Defect-related emission and distinctive absorption were detected by UV-Vis and PL spectroscopy, suggesting promise for optoelectronic uses. The nanoparticles' uniform morphology and polycrystalline nature were validated by TEM and HR-TEM investigations. The results show that hydrothermal synthesis is an easy, scalable, and environmentally friendly way to customize CoO nanoparticles for use in electrical devices, sensors, and catalysis. By encouraging innovation through sustainable nanotechnology, we can support SDG 9 by advancing nanotechnology for sustainable industrial innovation.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-9: Industry, Innovation and Infrastructure*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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