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Synthesis And Characterization Of Ni Metal, Cofe$_2$O$_3$, And Ni-Doped Cobalt Ferrite Using The Combustion Method

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This study reports the synthesis and characterization of nickel (Ni) metal, cobalt ferrite ($CoFe_2O_3$), and nickel-doped cobalt ferrite using the combustion method. The synthesis process employs metal carboxylates as precursors, with polyvinyl alcohol (PVA) acting as a fuel to facilitate combustion. The prepared samples were characterized using various techniques, including X-ray diffraction (XRD) for phase identification, scanning electron microscopy (SEM) for morphological analysis, Fourier transform infrared spectroscopy (FTIR) for functional group identification, and vibrating sample magnetometry (VSM) for magnetic property evaluation. The findings provide insights into the structural, morphological, and magnetic properties of the synthesized materials, highlighting their potential applications in electronic, biomedical, and catalytic fields.

KEYWORDS: PVA, cobalt ferrite ($CoFe_2O_3$), XRD, SEM.

Introduction

Nanomaterials serve as the cornerstone of nanoscience and nanotechnology, both of which are vast and interdisciplinary fields that have witnessed rapid advancements in recent years. Nanotechnology revolves around manipulating matter at the atomic and molecular scale, typically within the size range of 1–100 nm [1]. Recently, researchers have shown a growing interest in nanomaterials due to their remarkable properties and diverse applications [2]. The continual advancements in the physical properties and synthesis techniques of nanoscale materials have significantly contributed to the progress of nanoscience [3, 4].Human curiosity and the pursuit of knowledge have long driven the exploration of both the vast and the minuscule aspects of the physical world, encompassing mass, length, and time [5]. In some cases, the reduction in particle size to the nanoscale induces profound transformations, leading to the emergence of entirely new properties that revolutionize various fields [6,7]. One particularly intriguing aspect of nanotechnology is its impact on biological activity, as certain materials exhibit novel behaviors when reduced to nanoscale dimensions. However, amidst the rapid advancements in nanotechnology, one of the primary concerns remains the potential environmental impact of nanoparticles (NPs). A crucial method for assessing nanotoxicity involves monitoring bacterial responses to nanoparticle exposure [8–10].The applications of nanomaterials are vast and ever-expanding. Researchers are developing innovative uses such as attaching antibodies to nanotubes for bacterial detection, incorporating nanotubes into composite materials for aerospace applications, and using nanotubes infused with boron or gold to aid in oil spill cleanup. Other applications include miniaturized transistors, silicon-coated nanotube anodes that enhance lithium-ion battery capacity by up to tenfold, and nanotube-polymer nanocomposites designed as scaffolds to accelerate bone regeneration [11–15].

Among the various nanomaterials, transition metal ferrites—both doped and undoped—have emerged as promising candidates for a broad range of applications, including catalysis, sustainable hydrogen production, and electronic and magnetic devices [11–15]. In particular, cobalt ferrite ($CoFe_2O_4$) has garnered significant interest in both fundamental and applied research due to its exceptional mechanical hardness, thermal stability, and high anisotropy constant. These properties make cobalt ferrite highly suitable for applications spanning from medical technology to electronic devices. In light of these potential applications, numerous research groups have investigated the influence of rare earth cation doping on the properties of $CoFe_2O_4$ in bulk, thin-film, and nanoparticle forms [16].Cobalt ferrite has recently gained attention as a magnetostrictive material due to its promising magnetostrictive properties, making it a strong candidate for sensor and actuator applications. In these applications, rapid response times and precise displacement control are critical. While cobalt ferrite exhibits a relatively small magnetostrictive coefficient—which may be considered a drawback—it also possesses an extremely low hysteresis characteristic. This property minimizes energy loss and enhances displacement accuracy at high frequencies, thereby offsetting the disadvantage of a lower magnetostrictive coefficient. Consequently, cobalt ferrite holds significant potential for advancing next-generation sensor and actuator technologies[17].

In this study, cobalt ferrite nanomaterials and their nickel-doped (Ni-doped) nanocomposites were synthesized using the solid-state combustion method. Polyvinyl alcohol (PVA) was employed as a fuel to facilitate the combustion reaction. The structural characterization of the prepared cobalt ferrite and its Ag-doped nanocomposites was performed using X-ray diffraction (XRD), while their morphological analysis was conducted using Scanning Electron Microscopy (SEM). The results provide valuable insights into the structural and morphological properties of the synthesized nanomaterials, paving the way for their potential applications in biomedical and other technological fields.

Materials and Methodology

The following high-purity chemicals were used in this study:

Nickel nitrate ($Ni(NO_3)_2 \cdot 6H_2O$)
Cobalt nitrate ($Co(NO_3)_2 \cdot 6H_2O$)
Iron nitrate ($Fe(NO_3)_3 \cdot 9H_2O$)
Citric acid ($C_6H_8O_7$)

Polyvinyl alcohol (PVA) as a fuel
Deionized water

Synthesis of Ni, $CoFe_2O_3$, and Ni-Doped Cobalt Ferrite

1. **Preparation of Precursor Solution**

- Metal nitrates were dissolved in deionized water in stoichiometric ratios.
- Citric acid was added as a complexing agent to form metal carboxylates.
- The solution was continuously stirred until a homogeneous mixture was obtained.

2. **Combustion Process**

- PVA was added as a fuel to facilitate combustion.
- The solution was heated at 400–600°C in a muffle furnace to initiate self-propagating combustion.
- The reaction resulted in the formation of voluminous fine oxide powder

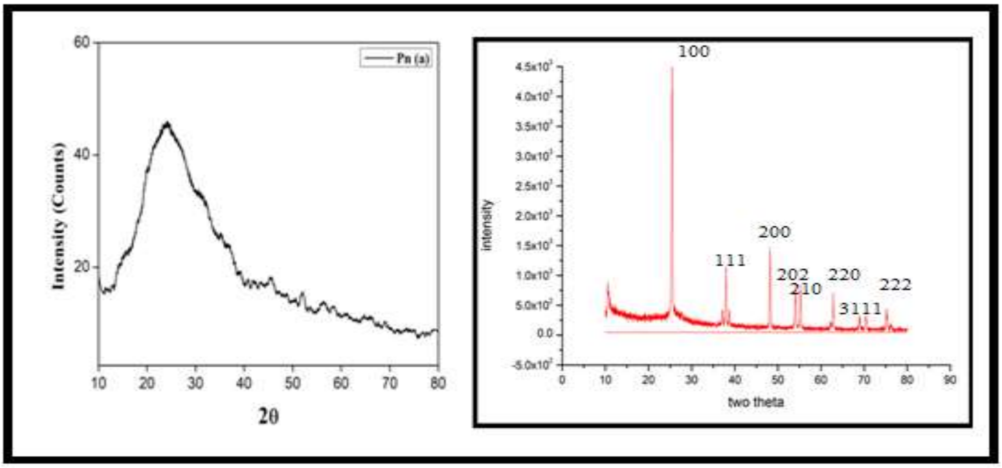
The synthesis of Ni metal, $CoFe_2O_3$, and Ni-doped cobalt ferrite ($Co_{1-x}Ni_xFe_2O_4$) using the combustion method involves a self-sustaining exothermic reaction. This method is widely used due to its ability to produce fine, homogeneous, and crystalline powders with controlled stoichiometry. The characterization of these materials is performed using various techniques such as X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), and AC/DC[18].

X-Ray Diffraction (XRD) Analysis

The XRD spectrum of polyaniline was recorded at a scan rate of 4° per minute, as shown in Figure [19].The XRD spectra of Cofe2o3 composite exhibits the diffraction peaks belongs to both NiO/$CoFe_2O_3$ and which describes the withholding of in the composite material. The XRD spectra of prepared Cofe2o3 composite exhibits well defined diffraction peaks obtained at different 2θ angles 37.42^0, 43.3^0, 62.34^0, 75.4^0 and 79.38^0 corresponding to the hkl planes (111), (200), (220), (311) and (222) for the phase of the nickel oxide and which are well matched with the reported literatures and standard JCPDS data card No. 73-1523 [13-14]. Previously it is reported (Arunkumar Lagashetty @al) defining the peaks of the NiO/$CoFe_2O_3$. The continuation to the previous work reported it is illustrated the determination of the average crystallite size of composite especially for all the composites of weight percentage 5. The average crystallite size was determined by using Scherrer's formula $D = K\lambda / \beta Cos\theta$ (where $\lambda = 1.54060$ θ is the Bragg angle, K is the Debye Scherrer constant and β is the peak full width at half maximum of the peak. The average crystallite size of Cofe2o3 composite was found to be 20nm[20].

Peak No.	2θ Position (°)	Miller Indices (hkl)
1	25.0	(100)
2	37.94	(111)
3	48.2	(200)
4	54.0	(202)
5	56.0	(210)
6	64.0	(220)

7	70.0	(311)
8	77.0	(222)



Fiure: XRD patterns of NiO/$CoFe_2O_3$

Scanning electron microscopy (SEM): Figure shows the SEM micrographs of synthesized NiO/$CoFe_2O_3$ composite. As prepared sample showed morphology with randomly distributed micro-sized round shaped particles with uniformity on the surface as well as a few agglomerations. The composite micrograph showed morphology slightly different from that of the micrograph of pure suggesting the possible presence of NiO/$CoFe_2O_3$ distributed in the matrix. The SEM image shows uniform morphology with semi-crystalline like structure and has a substantial intra-granular distance between the grains. The SEM micrographs of the composites shows highly agglomerated and irregular arranged granular in shape under different magnification. The grains are found to be well connected which suggests the higher binding energy between the grains. It is noticeable that the SEM micrograph of NiO/$CoFe_2O_3$ composites presents different morphology as compared with the ferrites[21].

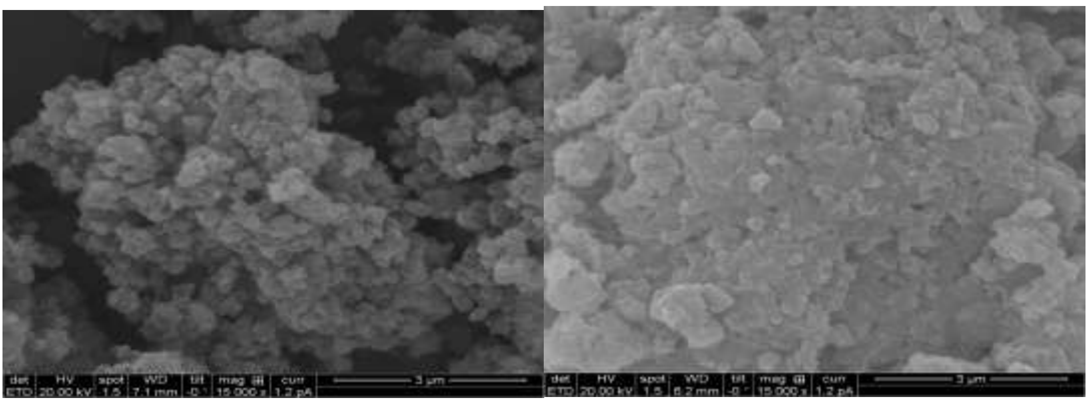


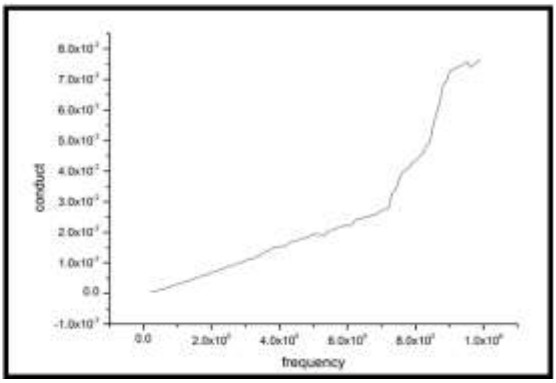
Figure: SEM micrographs of NiO/$CoFe_2O_3$

AC. conductivity

The A.C conductivity for the common polymer is changes with the frequency according to the following formula (Joshi & Sinha, 2007)[22].

A. $$\sigma_{ac}(\omega) = \sigma_t - \sigma_{d\tau} = A\omega^s$$

The frequency-dependent AC conductivity of NiO/$CoFe_2O_3$ is shown in Fig.. The conductivity of NiO/$CoFe_2O_3$ remains constant up to a frequency of $2x10^7$ Hz, after which it increases at higher frequencies [23-26]. The maximum conductivity seen in NiO/$CoFe_2O_3$ samples might be related to the chain length, as evidenced by the IR graph. Enhanced charge polarization might be the cause of increased conductivity



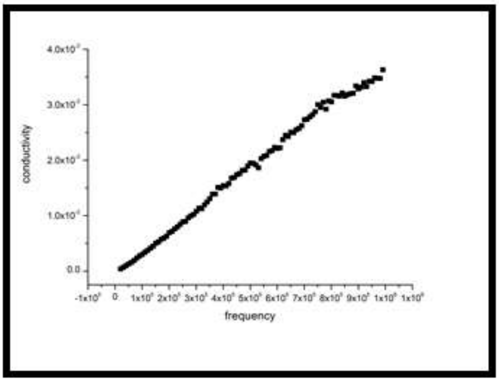


Figure: frequency-dependent AC conductivity of NiO/$CoFe_2O_3$

DCconductivity

The temperature-dependent DC electrical conductivity was investigated to understand the charge transport mechanism in the polymer composites. The variation in DC conductivity with temperature for the pure and composites is presented in Figure [28]. The two probe technique was used to describe the DC conductivity of the ferrite sample andeffect of NiO on electrical property of the composite. Figure is the DC conductivity asfunction of temperature of the NiO/$CoFe_2O_3$. Almost the same trend in the variation of the conductivity of the NiO and its composites was observed. It is observed that the value of dc conductivity of these composites increases exponentially with temperature due to increase in free charges with increase in temperature[27].The variation of dc conductivity as a function of temperature which recommends that as temperature increases conductivity also increases, hence thermally activated exponential behavior of conductivity has been observed. It remains nearly constant up to 100^0C and thereafter it increases exponentially.There is higher order increase in the conductivity at higher temperature phase and lowered conductivity at low temperature phase. The increase in the conductivity at higher temperature due excitation of electrons to the conduction band at higher temperature[29]. The conductivity behaviour is the characteristic of amorphousmaterials. The initial increase in the values of conductivity is due to the extended chain length of ferrites due to which the charge carriers can hopp between the favourable localized sites. The decrease in the values of conductivity of all the wt% of the composite is due to partial blocking of charge carriers. Further, gradual increase in conductivity is noticed due to the variation in distribution of $NiO/CoFe_2O_3$ particles in composite[27].

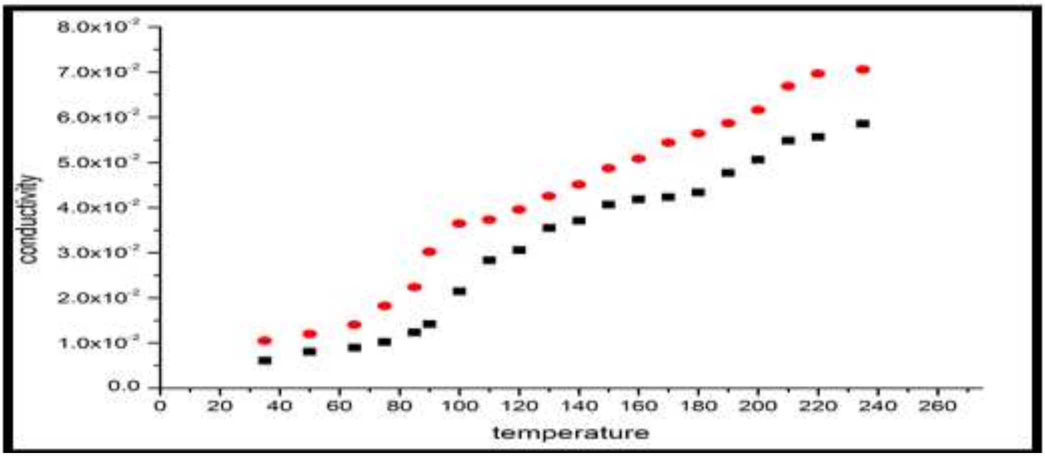


Figure: The DC conductivity of pure and composite

Conclusion

This study successfully synthesized Ni metal, $CoFe_2O_3$, and Ni-doped cobalt ferrite using the combustion method. The structural, morphological, and magnetic characterizations confirmed the formation of high-purity nanomaterials with enhanced properties. These findings highlight the potential applications of these materials in various technological fields, including electronics, magnetism, and biomedical applications.

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