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## Synthesis, Characterization and Structural Properties of $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$ /Polypyrrole Nanocomposites

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### Abstract

Polypyrrole (PPy) was synthesized via a chemical oxidative polymerization route using pyrrole monomer in the presence of different oxidizing agents. In parallel, hexagonal ferrite nanoparticles with the chemical composition  $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$  ( $x= 0.1, 0.2$  and  $0.3$ ) were successfully prepared using the sol-gel auto-combustion technique. Analytical-grade precursors, including nickel nitrate, cobalt nitrate, ferric nitrate, bismuth nitrate, and citric acid, were utilized without further purification. Stoichiometric quantities of the metal nitrates were accurately weighed and dissolved in ethylene glycol to produce a homogeneous salt solution. Citric acid was subsequently introduced as a chelating agent while maintaining a cation-to-citric acid molar ratio of 1:1.5. The pH of the resulting solution was adjusted to 7.0 through the dropwise addition of ammonia solution to facilitate gel formation and ensure uniform metal ion distribution prior to auto-combustion synthesis. The structural, morphological, compositional, and magnetic properties of the synthesized  $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$  nanocomposites were investigated using Fourier Transform Infrared Spectroscopy (FTIR), Field Emission Scanning Electron Microscopy (FESEM), and Energy Dispersive X-ray Spectroscopy (EDX). FTIR analysis confirmed the successful formation of the polymer-ferrite composite structure, while EDX spectra verified the elemental composition of the synthesized materials. FESEM micrographs revealed that the ferrite nanoparticles were uniformly and completely encapsulated by the polypyrrole matrix. Magnetic hysteresis measurements further demonstrated low coercivity values, confirming that the synthesized nanocomposites exhibit soft magnetic behavior suitable for advanced electromagnetic and electronic applications.

### Keywords

Polypyrrole,  
Ethylene glycol,  
FESEM,  
EDX,  
Nanocomposite  
Ammonia

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## 1. Introduction

Nanoparticles are materials that possess special optical properties and different physical properties which are desirable in technological process (Patange et al., 2010, Balavijayalakshimi et al., 2012, Singhal et al., 2012, Rani et al., 2018). The spinel ferrites have excellent magnet, electrical property and applications in high density microwaves and fluid magnet. Nickel ferrite ( $\text{NiFe}_2\text{O}_4$ ) is one of the most important spinel ferrites and has compositional spinel structure that begin from the magnetic moment of anti-parallel spins between the  $\text{Fe}^{3+}$  ions at tetrahedral structure and  $\text{Ni}^{2+}$  ions at octahedral structure (Patange et al., 2010). Nickel ferrite ( $\text{NiFe}_2\text{O}_4$ ) has wide applications in various fields in studying the electric and magnetic properties such as high saturation magnetization, stability, resistivity and low loss energy over a wide range of frequency. Various physical properties of nanocrystalline ferrites are highly influenced by distribution of cations among the sub lattices, nature of grain (shape, size, and orientation), grain boundaries, voids, inhomogeneity, surface layers and contacts. Due to their potential importance, many efforts to prepare high-quality magnetic nanocrystals of varying compositions have been stimulated (Yu Gao et al., 2019).

Bismuth and cobalt are the non-magnetic divalent metal ion doped with nickel ferrite. The substitution of Bismuth and cobalt in nickel ferrite enhances the properties of nickel ferrite, which are useful in many device applications. The doping materials are significant to magnetic materials due to their high electrical resistivity, high saturation magnetization and high magnetic permeability. Conducting polymer-based composites are novel materials with less than a decade of history. It is believed that the total control of the whole conducting polymer-based composite system and the optimisation of their physical properties

(such as electrical conductivity and colloidal stability) are yet to be achieved, while both their commercial availability in the near future and a big leap forward for materials science are expected with their appropriate utilization (Gangopadhyay & Amitabha, 2000).

In the case of biodegradable polymer-based nanocomposites, recent developments in preparation, characterization and properties, including crystallization behaviour and melt rheology, of both the matrix and the layered (montmorillonite) nanocomposites have been discussed (Pandey et al., 2005). Similarly, an emphasis on toughness and interfacial bonding between CNTs and polymer matrices is critically discussed to underline the stress transfer from the matrix and the potential of these composites for possible macro scale CNT-polymer production (Andrews & Weisenberger, 2004). Here, problems encountered so far are considered, and hints given regarding a critical volume fraction of CNTs to get appropriate strengthening (as observed in microcomposites); possible failure mechanisms in such composites were also presented. In view of the very limited work on metal-based nanocomposites including the ones with CNT reinforcements, no review is available to-date on this system. Considering these facts and also the absence of a more general report comprising the three different kinds of nanocomposites (metal-, ceramic- and polymer-based), including those with incorporation of CNTs (Schmidt et al., 2002, Braun et al., 1997, Biercuk et al., 2002, Ounaies et al., 2003, Weisenberger et al., 2003, Choa et al., 2003).

Inorganic-organic nanocomposite materials have attracted more and more attention as they introduce new materials, which can combine different functions and characteristics of individual constituents

(Irshad et al. 2013). In microwave applications, ferrite-polymer composites may serve as better electromagnetic interference (EMI) suppressors (Irshad et al. 2013, Junhai et al. 2014). And among different polymers, conducting polymers with their good electrical conduction and high dielectric loss factor may play a crucial role in the microwave absorbing applications (Saafan et al. 2011). In this context, polypyrrole (PPy) is one of the most promising conducting polymers for potential applications in electronic devices on the basis of its conductivity, excellent environmental stability, relatively low density and ease in synthesis (Saafan et al. 2011). PPy as a new material has opened up an entirely new field for polymeric materials. Therefore, many interesting types of researches focus on ferrite/PPy composites to get functional materials with the characteristics of both conductive polymer and ferrite nanoparticles (Wang et al. 2008, Dubal et al. 2011, Xie et al. 2012, Penghui et al. 2013).

In the present work, the sample of  $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$  has been chosen from the series of Co-Ni-Li nanoferrites (Assar et al. 2014, Ninad et al. 2019), because of its best electromagnetic properties as mentioned above to prepare and study the structural, magnetic and electrical properties of  $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$ /PPy nanocomposites with different weight ratios. Many Researchers have focused their attention on Ni-Bi-Co ferrites because Bismuth substituting ferrites have different and interesting magnetic and electrical properties (Saafan et al. 2011). Several research groups have reported that, with that Bi material, magnetization in Ni-Bi ferrites decreases and vice versa. Several research groups have also stated that magnetization in Ni-Co ferrites decreases as Co content increases. A number of Researchers have been reported that the

studying the effect of composition on the electromagnetic wave absorption properties. In the present task, we have employed sol-gel auto combustion method to investigate the effect of  $\text{Bi}^{2+}/\text{Co}^{2+}$  ion substitution on structural, magnetic and physical properties of  $\text{NiFe}_2\text{O}_4$  nano-crystalline ferrite (Babu et al., 2018).

## 2. Experimental procedure

### 2.1 Materials and apparatus

The reagents and solvents of high purity were purchased from Merck, Fluka, and Aldrich Chemical Companies. The used ethylene glycol in chemical reactions was purified by the standard method. Fourier transform infrared (FT-IR) spectra were obtained as potassium bromide pellets with Thermo Nicolet IMPACT-400 FT-IR spectrophotometer in  $400\text{-}4000\text{ cm}^{-1}$ . The field emission scanning electron microscope (FE-SEM) of the surface, energy dispersive X-ray spectroscopy (EDX).

### 2.2 Procedure for preparation of $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$

Nanocrystalline Bismuth doped Nickel ferrite and Cobalt doped Ferrites  $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$ . Desired composition was obtained by using stoichiometric amounts of Nickel Nitrate, Bismuth Nitrate, Cobalt Nitrate and Ferrite Nitrate dissolved in 50 mL distilled water. And also used suitable amounts of citric acid which is Polymer for combustion. The neutralization was carried out by adding 10 mL of ethylene glycol and 10 mL hydrogen peroxide. The solution was heated on a hot plate with constant and continuous magnetic stirring at  $80\text{-}100\text{ }^\circ\text{C}$  in to evaporate the water. A brown gel was obtained which upon further heating at  $280\text{-}300\text{ }^\circ\text{C}$  gives the precursor material. The precursor material was calcinated at  $1000\text{ }^\circ\text{C}$  for 6 hours.

Table 1. Molar ratios of reagents for A1 preparation

	Mole Ratio				Polymer	Samples
<b>Mole Ratio</b>	Ni(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (0.9)	BI(NO <sub>3</sub> ) <sub>3</sub> ·5H <sub>2</sub> O (0.1)	Fe(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (1.9)	Co(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (0.1)	Citric acid	Ni <sub>0.9</sub> Bi <sub>0.1</sub> Fe <sub>1.9</sub> Co <sub>0.1</sub> O <sub>4</sub>
<b>Molecular weight (MW)</b>	290.8	485.07	404	291.03	210.14	
<b>MW * Mole Ratio</b>	261.72	48.507	767.6	29.103	630.42	
<b>Amount (g)</b>	10.4688	1.94028	30.704	1.164	25.217	A 1

Table 2. Molar ratios of reagents for A 2 preparation

	Mole Ratio				Polymer	Samples
<b>Mole Ratio</b>	Ni(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (0.8)	BI(NO <sub>3</sub> ) <sub>3</sub> ·5H <sub>2</sub> O (0.2)	Fe(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (1.8)	Co(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (0.2)	Citric acid	Ni <sub>0.8</sub> Bi <sub>0.2</sub> Fe <sub>1.8</sub> Co <sub>0.2</sub> O <sub>4</sub>
<b>Molecular weight (MW)</b>	290.8	485.07	404	291.03	210.14	
<b>MW * Mole Ratio</b>	232.64	97.014	727.2	58.2	630.42	
<b>Amount (g)</b>	9.306	3.880	29.088	2.328	25.217	A 2

Table 3. Molar ratios of reagents for A 3 preparation

	Mole Ratio				Polymer	Samples
<b>Mole Ratio</b>	Ni(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (0.7)	BI(NO <sub>3</sub> ) <sub>3</sub> ·5H <sub>2</sub> O (0.3)	Fe(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (1.7)	Co(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O (0.3)	Citric acid	Ni <sub>0.7</sub> Bi <sub>0.3</sub> Fe <sub>1.7</sub> Co <sub>0.3</sub> O <sub>4</sub>
<b>Molecular weight (MW)</b>	290.8	485.07	404	291.03	210.14	
<b>MW * Mole Ratio</b>	203.56	145.521	686.8	87.3	630.42	
<b>Amount (g)</b>	8.142	5.821	27.472	3.492	25.217	A 3

Where amount = Molecular weight (MW) \* Mole Ratio / 25

### 2.3 Procedure for the Synthesis of Polypyrrole

Pyrrole is a five membered heterocyclic compound, with corresponding general formula  $C_4H_4NH$ . It is a colourless volatile liquid, unstable in air, where it easily darkens. Polypyrrole (Ppy) was synthesized by chemical oxidative polymerization technique using pyrrole monomer and different oxidants. The chemical polymerization was carried out in a beaker with 100 ml of hydrochloric Acid by mixing with pyrrole monomer and ammonium persulphate.

Different concentrations of pyrrole monomer and ammonium persulphate were used. Sample IDs A2 and A3 were mixed with polypyrrole (prepared in-house) to form a composite. About 2.5 g of A2 was taken in a watch glass. 0.5 g of polypyrrole (i.e. 10% by weight of A2) (prepared in-house as described in sec. 3.3.2) was weighed in another watch glass. These two materials were transferred into mortar and pestle (ceramic). Mixture was grounded manually using a pastel for ~ 10 minutes. This sample (i.e. the composite) was named as AA2. Again about 5 g of A3 was taken in a watch glass. 1 g (i.e. 20% by weight of A3) of polypyrrole was taken in another watch glass. These two materials were transferred in a mortar and pestle (ceramic). Mixture was then grounded thoroughly manually for ~ 10 minutes. This sample (i.e. the composite) was named as AA3.

## 3. Results and discussions

### 3.1 Preparation and characterization of $Ni_{0.9}Bi_{0.1}Fe_{1.9}Co_{0.1}O_4/PPy$

The composite of Nickel ferrites ( $Ni_{0.9}Bi_{0.1}Fe_{1.9}Co_{0.1}O_4$ ) with polypyrrole have been successfully synthesized. After preparation of the catalyst and composite, various techniques including Fourier-

transform infrared spectroscopy (FT-IR), field emission scanning electron microscope (FESEM), energy dispersive X-Ray analysis (EDX), were used to study and identify the structure, morphology, and functional groups. The FTIR shown the stretching vibrations of tetrahedral and octahedral parts of A1, A2 and A3 composites. Moreover, the peaks observed in A2 and A3 at around  $1000\text{ cm}^{-1}$  confirmed the presence of polypyrrole (Ppy) which is not present in A1. The FESEM micrograph and EDX spectrum indicated the presence of Ni, Bi, Co, C, N, and Fe and O ions with no significant impurity in the prepared ferrites and confirms the purity of the final products.

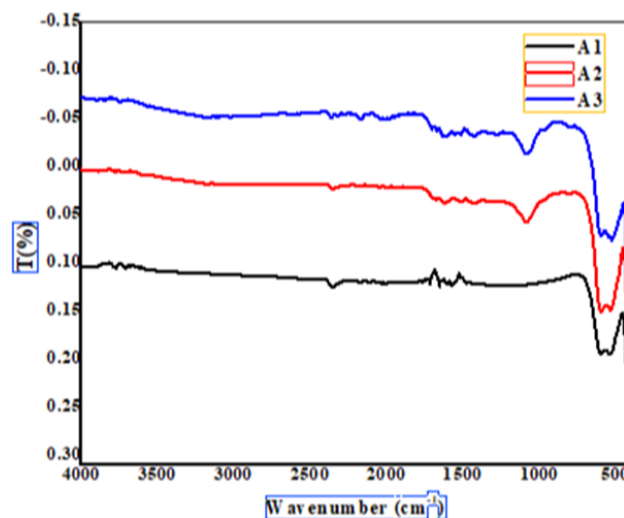
### 2.4 Procedure for the formation of doped Ni-ferrites/polypyrrole composite

prepared, in the ratio of 2.4 gm of ammonium persulphate to 1 ml of pyrrole (therefore, 3.13 ml of pyrrole was mixed with 7.512 gm of Ammonium persulphate). Since this is an exothermic reaction, the addition of distilled water (cooled) was done slowly. And vigorous magnetic stirring was maintained to facilitate the dispersion of pyrrole. The polymerization reaction was carried out for a period of 4 hrs at a temperature of  $30\text{ }^\circ\text{C}$ . The polymer solution was filtered and washed several times with distilled water. The black PPy powder obtained was dried for 8 hours in an oven at  $60\text{ }^\circ\text{C}$  overnight.

FTIR analysis shows the metal oxygen vibration modes present at the ferrite system's tetrahedral and octahedral sites are further confirmed using FTIR spectra. Fig 1 represented the corresponding metal oxygen vibrations and functional groups present in the magnetic  $Ni_{0.9}Bi_{0.1}Fe_{1.9}Co_{0.1}O_4$  ferrite nanoparticles with polypyrrole. It can be observed that there is a weak peak at  $600\text{ cm}^{-1}$  corresponding to vibrations of tetrahedral and octahedral sites for A1, A2

and A3. However, the peak that cannot be observed in A1 composite is almost identified by the characteristic peaks of PPy. The peaks between 900-1050  $\text{cm}^{-1}$  are attributed to the C-H in-plane bending and the ring deformation. The peaks around 1500  $\text{cm}^{-1}$  cause by the C-N and C=C stretching vibrations. The peaks at 1210  $\text{cm}^{-1}$  correspond

to N-H in-plane deformation vibrations. Moreover, the peaks at 1610  $\text{cm}^{-1}$  (O-H bending vibration) and 3420  $\text{cm}^{-1}$  (O-H stretching vibration) suggested that there is some water in samples. The results indicate that PPy was successfully introduced to nanocomposite (Hosseini & Asadnia, 2012, Liu et al., 2019).



**Figure 1** FTIR spectra of  $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$ /Polypyrrole nanocomposites (a) A1, (b) A2, and (c) A3

The Field Emission Scanning Electron Microscopy (FESEM) Produced by electrochemical polymerization in Figure 2 shows that the particles are irregular in cubic shape. The micrographs shown the presence of spherical grains with distribution of heterogeneous size and changing the degree of agglomeration. The formation of agglomerated grain structure is a characteristic feature of combustion synthesis method, and the willingness of nanomaterial agglomeration is a direct

consequence of enhancing surface energy and small grain sizes. The average particle size measured by increase FESEM images using sampling techniques for Cobalt doping are 49 and 52 nm for A1 and A2 respectively. Perhaps dosage of pyrrole is low, causing low adhesion to the surface of the PPy and  $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$  composites. With dosage of pyrrole increasing, there is no shedding between PPy shell and  $\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4$  composites (Hosseini & Asadnia, 2012, Luo et al., 2013, Liu et al., 2019).

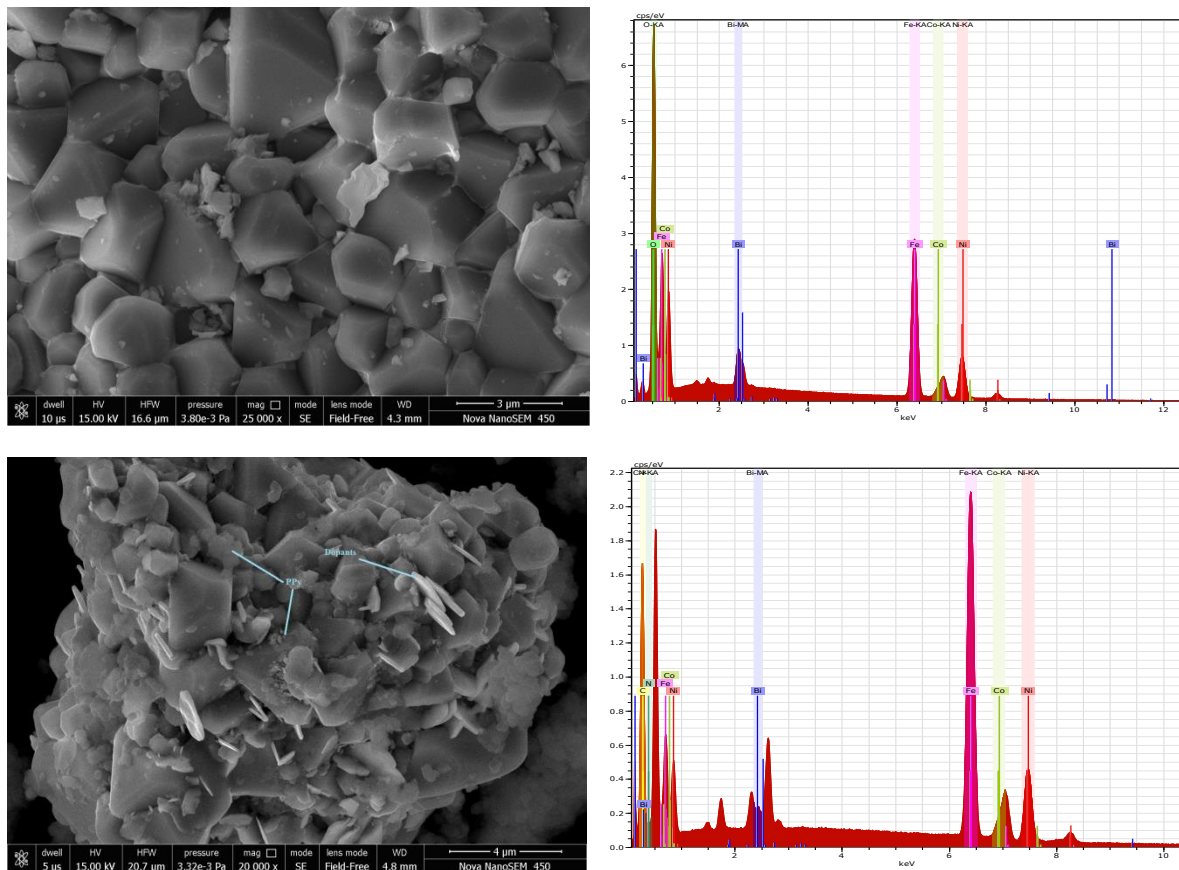


Figure 2 FESEM/EDX of A1 and A2

#### 4. Conclusion

Nickel ferrite/polypyrrole nanocomposites with the composition  $(\text{Ni}_{0.9}\text{Bi}_{0.1}\text{Fe}_{1.9}\text{Co}_{0.1}\text{O}_4)$  were successfully synthesized using the sol-gel auto-combustion method followed by chemical oxidative polymerization. The synthesized composites were effectively characterized using Fourier Transform Infrared Spectroscopy (FTIR), Field Emission Scanning Electron Microscopy (FESEM), and Energy Dispersive X-ray Spectroscopy (EDX) to evaluate their structural, morphological, and compositional properties.

The FTIR spectra revealed characteristic stretching vibrations corresponding to the tetrahedral and octahedral sites of the ferrite lattice in A1, A2, and A3 composites, confirming the successful formation of the spinel ferrite structure. Furthermore, the appearance of distinct absorption peaks around  $1000\text{ cm}^{-1}$  in A2 and A3

clearly confirmed the incorporation and successful coating of polypyrrole (PPy) within the ferrite matrix, whereas these peaks were absent in A1, indicating the nonexistence of PPy in that sample.

Morphological analysis obtained from FESEM micrographs demonstrated that the ferrite nanoparticles were uniformly coated and embedded within the PPy matrix, resulting in improved surface homogeneity and particle distribution. In addition, EDX spectra confirmed the presence of Ni, Bi, Co, Fe, O, C, and N elements without detectable secondary phases or significant impurities, thereby establishing the high purity and successful synthesis of the nanocomposites. The combined structural and compositional analyses indicate that the prepared ferrite/PPy nanocomposites possess promising characteristics for potential applications in soft magnetic materials, electromagnetic devices, sensors, and advanced electronic systems.

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