Baku State University: Journal of Physics & Space Sciences, **2026**, v3 (2), pp. 54-62

journal homepage: http://bsuj.bsu.edu.az/en

Structural properties of polypropylene nanocomposites reinforced with cobalt ferrite nanoparticles

[1]Ayshan Z. Guliyeva*, [2]Flora V. Hajiyeva, [3]Rana F. Babayeva, [2]Nushaba O. Gadirova

[1]Department of Physical Electronics, Baku State University, Baku, Azerbaijan
[2]Department of Chemical Physics of the Nanomaterials, Baku State University, Baku, Azerbaijan
[3]Department of Engineering and Applied Sciences, Azerbaijan State University of Economics, Baku, Azerbaijan

Received 01-Apr.-2026; Accepted 15-May.-2026
DOI: https://doi.org/10.30546/209501.101.2026.3.02.627

Abstract

In the present study, cobalt ferrite nanoparticles were synthesized and stabilized, and novel polypropylene-based nanocomposites were subsequently fabricated on their basis. The structures of the nanoparticles and nanocomposites were investigated using X-ray diffraction (XRD), transmission electron microscopy (TEM), and infrared (IR) spectroscopy analyses. XRD analysis demonstrated that peaks corresponding to $CoFe_2O_4$ nanoparticles were not observed at low nanoparticle concentrations within the polymer matrix. As the concentration of nanoparticles increased, characteristic peaks of $CoFe_2O_4$ nanoparticles became evident, accompanied by an increase in the degree of crystallinity of the PP + $CoFe_2O_4$-based nanocomposites. IR analysis revealed that no new peaks appeared upon the incorporation of $CoFe_2O_4$ nanoparticles into the system, indicating the absence of chemical interactions. Only an increase in the intensity of the absorption bands was observed, suggesting the presence of physical interactions.

Keywords: cobalt ferrite, chemical precipitation, spinel structure, magnetic properties
PACS Numbers: 11.55.Jy

*Corresponding author – Tel.: (+994) 70 719 03 89
e-mail address: ayshan.quliyeva@bsu.edu.az; *ORCID ID*: 0009-0003-2985-7171.

1. Introduction

Nanocomposite materials prepared from magnetic nanoparticles have gained considerable importance in modern materials science and nanotechnology. These magnetic ferrites with a spinel structure have attracted significant attention both in the global industrial market and among numerous researchers owing to their remarkable electromagnetic properties. Such ferromagnetic nanoparticles are dispersed within polymer nanocomposite materials and are distributed in the form of ferromagnetic monodomains. The presence of highly concentrated cobalt ferrite nanoparticles opens new perspectives for the development of high-density data recording materials. These magnetic nanoparticles can be successfully synthesized under various conditions, thereby offering broad opportunities for industrial applications. The ability to tailor their physical, chemical, electrical, and magnetic properties through modification of their constituent elements has encouraged scientists to develop materials with novel structures and enhanced functional characteristics [1].

The magnetic, magnetostrictive, and electronic functional properties of cobalt-rich ferrites have made them one of the most widely utilized ferrite systems in magnetic recording technologies and the production of magnetic devices. Spinel ferrites, particularly cobalt ferrite ($CoFe_2O_4$) nanoparticles, possess an inverse spinel structure and are considered highly suitable magnetic materials due to their high magnetocrystalline anisotropy, moderate saturation magnetization, high Curie temperature, favorable electrical properties, high mechanical strength, and chemical stability. Furthermore, particle size has a significant influence on magnetic properties. In nanoscale particles, superparamagnetism is observed as a result of surface effects and thermal fluctuations. As the particle size increases, a multidomain structure is formed, leading to changes in magnetic hysteresis characteristics [2–4]. Extensive research has been devoted to the preparation of cobalt ferrite using various methods. The most commonly employed techniques for synthesizing these highly magnetic nanoparticles include chemical precipitation, sol-gel synthesis, solid-state reaction, ceramic methods, thermal decomposition, hydrothermal synthesis, and others. Among these approaches, chemical precipitation is one of the most widely used wet chemical methods for cobalt ferrite synthesis due to its simplicity, low cost, ability to produce sufficiently small particles, and provision of good stoichiometric control. In this method, metal salts are dissolved in an appropriate solvent according to stoichiometric ratios. Subsequently, a precipitating agent is added to adjust the pH of the medium to the desired level, resulting in the formation of a single-phase, homogeneous inorganic solid. Through proper control of synthesis parameters, particle size, morphology, and magnetic properties can be optimized. However, additional heat treatment may be required because of aggregation and crystallinity-related issues [5].

One of the promising directions in materials science is the application of polymer-based nanocomposites. Such polymer-based nanocomposite materials exhibit excellent mechanical and optical properties, as well as tunable electrical and magnetic characteristics [6]. These materials are obtained by dispersing nanoscale inorganic or organic particles within a polymer matrix and combine the properties of both the polymer and the nanoparticles [7]. Owing to the high surface area of the incorporated nanoparticles, these nanocomposites demonstrate enhanced mechanical, electrical, thermal, and magnetic properties [8]. Polymer-based magnetic materials can be utilized in various fields, including medicine, magnetic recording systems, information technologies, and others [9]. Two methods are predominantly employed in the preparation of mineral-polymer nanocomposites formed through the dispersion of nanoparticles within a polymer matrix: blending pre-synthesized magnetic nanoparticles with a polymer and the in situ synthesis of nanoparticles within the polymer matrix. The primary objective is to employ a polymer matrix containing functional groups capable of forming chelates with metal ions. Polyvinyl alcohol (PVA) is one of the most suitable polymers for this synthesis approach. Ans, A.R. et al. demonstrated that the chemical, structural, and magnetic properties of PVA/$CoFe_2O_4$-based nanocomposites provide extensive opportunities for their application in remote actuation and sensing technologies in the biomedical sector [10–11].

Nanocomposite materials formed through the homogeneous dispersion of cobalt ferrite nanoparticles within a polystyrene polymer matrix exhibit high saturation magnetization and coercivity, together with excellent thermal and mechanical stability. The magnetic properties of PS/$CoFe_2O_4$-based nanocomposites are superior to those of $CoFe_2O_4$ nanoparticles synthesized under identical conditions. This improvement is attributed to the particle size and degree of crystallinity of the nanoparticles dispersed within the polystyrene matrix. In addition, the biocompatibility of polystyrene enables the broad application of these nanocomposites in medical fields [12–14].

Anna, C. et al. demonstrated that PLA/$CoFe_2O_4$-based nanocomposites exhibited a high recovery ratio (>84%) and showed rapid heating capability under the influence of a high-frequency magnetic field [15-16].

In the present study, nanocomposites based on polypropylene and cobalt ferrite nanoparticles were synthesized, and their structures were investigated using X-ray diffraction analysis and infrared spectroscopy techniques.

2. Experimental section

2.1. Synthesis of cobalt ferrite nanoparticles

Cobalt ferrite nanoparticles were synthesized using the alkaline co-precipitation method developed by Massart in the 1980s. In this method, metal precursors (Co^{2+} and Fe^{3+} ions) are dissolved in an acidic medium and subsequently co-precipitated in an alkaline medium, thereby preventing the formation of amorphous hydroxides.

Depending on experimental parameters such as pH, the type of base used, and the ratio of the reactants, this method allows precise control over particle size [17–18].

For the synthesis of cobalt ferrite ($CoFe_2O_4$) nanoparticles by the chemical precipitation method, 0.4 M $FeCl_3 \cdot 6H_2O$ and 0.2 M $CoCl_3 \cdot 6H_2O$ solutions were prepared separately in a molar ratio of 2:1 and dissolved individually in 50 mL of distilled water. Subsequently, the two solutions were combined, and 0.5 g of polyethylene glycol (PEG) was added as a stabilizing agent. The resulting mixture was stirred on a magnetic stirrer at 80°C for 15 min.

In the next stage of the experiment, a solution containing 6 g of NaOH dissolved in 50 mL of distilled water was gradually added to the previously prepared mixture, and the system was further stirred on a magnetic stirrer for 45 min. Upon completion of the reaction, the obtained nanoparticles were washed three times with ethanol to remove residual reagents and centrifuged at 5000 rpm for 5 min during each washing step. The synthesized samples were subsequently dried at 400°C for 60 min.

2.2. Synthesis of nanocomposites

The PP + $CoFe_2O_4$ nanocomposites were synthesized using the ex situ method. For this purpose, 0.5 g of PP polymer granules were dissolved in 50 mL of toluene at 110°C. Various weight fractions of $CoFe_2O_4$ magnetic nanoparticles were then added to the dissolved polymer system, and the mixture was stirred on a magnetic stirrer at 80°C for 30–40 min.

Subsequently, the homogeneous mixture was transferred into a Petri dish and dried at room temperature for several days. The polymer–nanoparticle bulk samples were further dried in a vacuum oven for 1 h to ensure complete evaporation of toluene from the polymer volume. The resulting bulk materials were then processed into thin films by hot pressing at the melting temperature of PP (160°C) under a pressure of 10 MPa, followed by water cooling.

2.3. Characterization

The diffraction patterns of the samples were recorded on a Rigaku Mini Flex 600 diffractometer. The IR spectra were analyzed on an Varian 3600 FT-IR spectrometer. Transmission electron microscopy (TEM) analysis was performed using a JEM-1400 transmission electron microscope, JEOL, Japan.

3. Results and discussion

Figure 1 shows the X-ray diffraction pattern of $CoFe_2O_4$ nanoparticles. As can be seen from the figure, the diffraction pattern contains peaks at 2θ angles of 30.360, 35.920, 43.680 and 57.780, corresponding to the spinel structure of cobalt ferrite.

Figure 1 shows also the diffraction patterns of PP+$CoFe_2O_4$-based nanocomposites. Polypropylene exhibits several diffraction peaks at 14° (110), 17° (040), 18.5° (130) and 21° (111) in the 2θ angle range from 5 to 30°. These peaks indicate the presence of predominantly monoclinic α-form in polypropylene. X-ray diffraction analysis revealed no peaks corresponding to the beta (β) and gamma (γ) phases in polypropylene (PP). It was found that at low nanoparticle content (1%) in the polymer, peaks corresponding to cobalt ferrite nanoparticles are not observed. With a further increase in the nanoparticle content (3.5, 10%), peaks corresponding to $CoFe_2O_4$ nanoparticles are observed, and the degree of crystallinity of nanocomposites based on PP+$CoFe_2O_4$ increases slightly. The average crystallite size was 9.3 nm. Thus, according to X-ray diffraction analysis, cobalt ferrite nanoparticles can act as effective nucleating agents, promoting heterogeneous nucleation of polymer chains and the formation of more ordered crystalline structures.

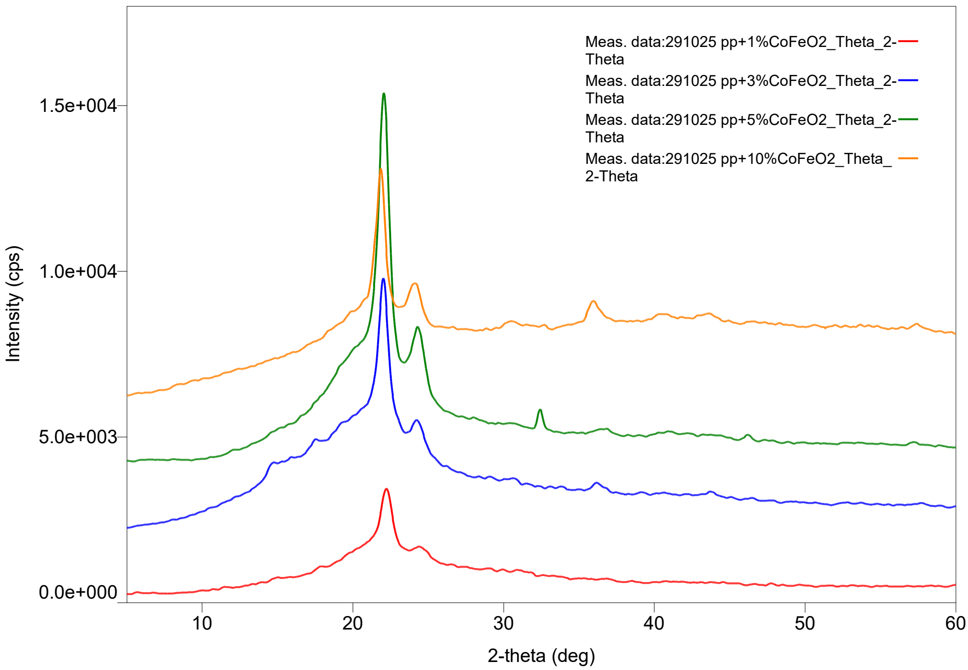


Fig. 1. Diffraction patterns of PP+$CoFe_2O_4$ nanoparticles.

The figure shows TEM images of $CoFe_2O_4$ nanoparticles. As can be seen from the images, the nanoparticles form a chain structure and have an average size of 10 nm.

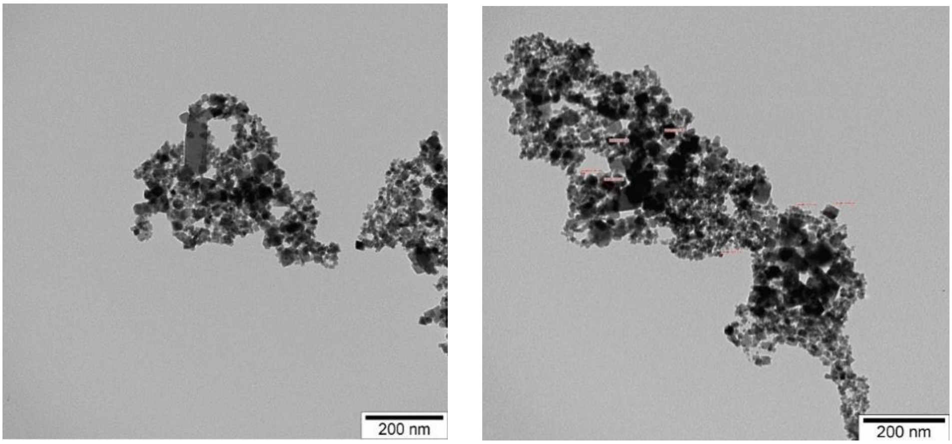
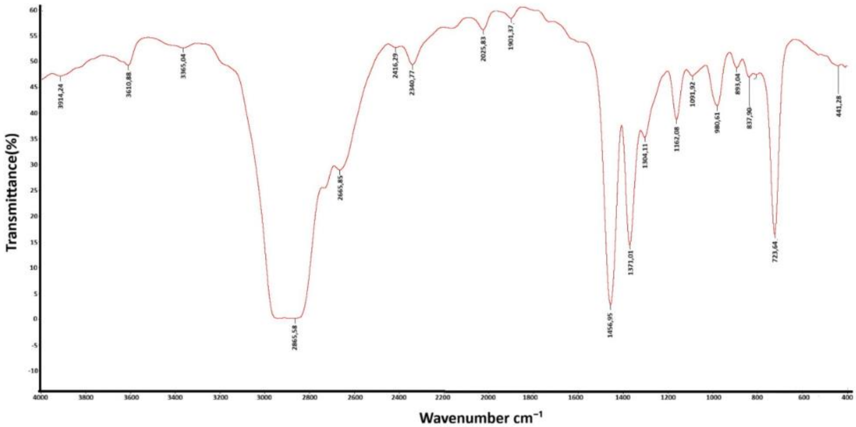
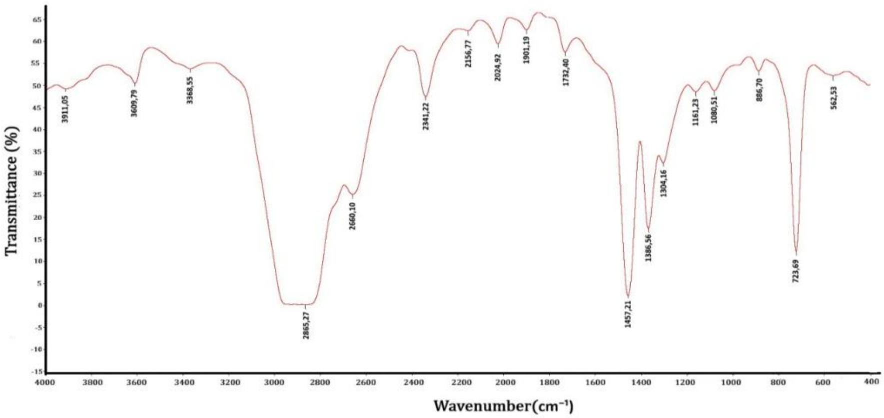


Fig. 2. TEM image of $CoFe_2O_4$ nanoparticles.



a)



b)

Fig. 3. Infrared spectra's of PP (a) and PP+5%CoFe2O4 based nanocomposites.

Figure 3 (a) shows the IR spectrum of pure isotactic polypropylene. A peak appears in the wavenumber range 3000–2800 cm^{-1}: the peak at 2865 cm^{-1} is attributed to the symmetric stretching vibrations of the CH_3 group, respectively. Two intense peaks are observed at wavenumbers 1456.95 and 1371.01 cm^{-1}: the peak at 1456.95 cm^{-1} corresponds to the antisymmetric deformation vibrations of the CH_3 group or the “scissors” vibrations of the CH_2 group, and the peak at 1378 cm^{-1} corresponds to the symmetric deformation vibrations of the CH_3 group. The peak at 1304 cm^{-1} is attributed to the rotation or plane vibrations of the CH_2 group about its axis. The peak at 1162.08 cm^{-1} corresponds to the C–C antisymmetric stretching, the antisymmetric cradle vibrations of CH_3, and the up-down synchronous vibrations of CH. The peak at 893.04 cm^{-1} is attributed to the deformation of CH groups or the cradle vibrations of CH_3 groups. The peaks at 837, 90 and 805.92 cm^{-1} are associated with the cradle vibrations of CH_2 group. The 723 cm^{-1} observed in the range of 700–1200 cm^{-1} is due to the interaction of the cradle vibrations of CH_2 and CH_3 groups with the stretching vibrations of the carbon skeleton. The absorption bands in the range of 500–150 cm^{-1} correspond to the deformation vibrations of the skeleton and the internal rotation movements of the chain. Figure 3 (b) shows the IR spectrum of the PP+5% CoFe2O4 nanocomposite. Here, a slight increase in the intensities of the peaks at 1456.95 cm^{-1} and 723 cm^{-1} belonging to polypropylene, and a decrease in the peaks at 2865.58 and 1371.01 cm^{-1} were observed. In addition, a peak appeared at 562 cm^{-1}, and this peak corresponds to the characteristic stretching vibrations of the metal–oxygen (Co–O) bonds in the tetrahedral position. The increase and decrease in the intensities of absorption bands in the FTIR spectra of PP/$CoFe_2O_4$ nanocomposites is due to the interfacial interaction between polypropylene macromolecules and the surface of cobalt ferrite nanoparticles, as well as changes in the degree of crystallinity and local area of the polymer's functional groups.

The mechanism behind these changes can be explained as follows: $CoFe_2O_4$ nanoparticles have a high specific surface area and create an interfacial layer between the filler and the polymer matrix. Within this layer, the orientation and mobility of the polypropylene macromolecular chains changes. As a result, some vibrations are enhanced due to increased structural ordering, while others are weakened due to limited mobility of functional groups and changes in the crystallinity of the nanocomposite [19-20].

Conclusion

IR analysis of nanocomposites shows that a polymer layer with reduced chain mobility is formed in the interfacial region, which causes a change in the dipole moment during vibrations of chemical bonds, which leads to a change in the intensity of absorption bands in the FTIR spectra. XRD analysis showed that cobalt ferrite nanoparticles can act as effective nucleating agents, leading to heterogeneous nucleation of polypropylene chains and the formation of more ordered crystalline structures [21-22].

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