

Synthesis and Characterization of Cobalt Ferrite through Co-Precipitation Technique

Subramanian Nithiyantham^{1,*} , Selvaraj Viviliya¹ , Somasundaram Anandhan¹ ,
Sreepathi Mahalakshmi² 

¹ Post Graduate & Research Department of Physics, (Applied Energy Resource Divisions), Thiru. Vi. Kalyanasundaram Govt Arts and Science College, Thiruvavur, Tamilnadu, India

² Post Graduate & Research Department of Physics, Ethiraj College for Women, Chennai, Tamilnadu, India

* Correspondence: s_nithu59@rediffmail.com;

Scopus Author ID 34768710400

Received: 4.08.2020; Revised: 20.09.2020; Accepted: 24.09.2020; Published: 3.10.2020

Abstract: The device formation in current technology demands effective magnetic materials. Cobalt ferrite nanoparticles were synthesized by the co-precipitations method using the precursor materials ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) and ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$). X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) analysis, and UV-Visible absorption spectral studies were used to analyze the structural, chemical/functional groups with possible stretching and optical bandgap properties of the CoFe_2O_4 powder. XRD results designate that the resultant particles are crystalline, pure single-phase spinel structure. From the FTIR analysis reveals that C-C, C=O stretching, and the shift is leaked indicating that the presence CoFe_2O_4 . The absorption and the optical band gaps values are increased trend with temperatures also evidence that is enhancing magnetic behavior.

Keywords: ferrite; XRD; FTIR; UV; bandgap.

© 2020 by the authors. This article is an open-access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Cobalt with magnetite plays a reasonable role in insensing processes such as chemical sensors, thermal sensors, magnetic sensors and etc. The peculiar property of the anisotropic nature of cobalt with Fe the susceptibility values exist in less one. But enhancing Co gives the susceptibility that goes greater than one may be used for novel sensing purposes [1-4]. Co_3O_4 is an antiferromagnet ($T_N \approx 30\text{K}$). In recent times many researchers working in the derivative compound of $\text{Co}_{3-x}\text{M}_x\text{O}_4$ [$\text{M} = \text{Fe}, \text{Al}, \text{Mn}$] [5]. The properties of compounds with Fe give variety/diverse applications through different magnetic nature such as para, ferro, anti-ferro, and ferric natures [6]. Spinel ferrite (MFe_2O_4) magnetic nanocomposites have fascinated much responsiveness owing to their distinctive magnetic assets and chemical stability. There are many ways of preparing nanoparticle with many ways sol-gel [7], co-precipitation [8] and etc.,

In this investigation, the author focuses on spinel, mainly spinel stability, with a range of temperatures (above 900°C it gradually decreases). The isotropic behavior of Co ion can change depends on the size and the nature of preparation processes [9]. To achieve this, we have synthesized cobalt ferrite magnetic nanoparticles through the co-precipitation method and characterized by using X-ray diffraction (XRD), Fourier transforms infrared (FT-IR), and UV-visible (UV-vis) spectroscopy through which determined the optical band gap of cobalt ferrite magnetic nanoparticles [10].

2. Materials and Methods

2.1. Sample preparation.

The nanoparticle Co_2FeO_4 were prepared by the chemical co-precipitation method [8]. The precursor material used was Iron (III) nitrate with Cobalt(II) nitrate was taken by 1:2 ratio of AR grade chemicals procured from S.D. Fine chemicals, Mumbai, India (Table.1). The mixed solution stirred well using a magnetic stirrer about 6 hr with temperature 80°C and maintained their pH (11) by the addition of Potassium hydroxide (KOH). Finally, the sample precipitate was found at the bottom of the beaker.

Table 1. Properties of the precursor material.

Sample	Molecular weight(g/mol)	Melting point ($^\circ\text{C}$)	Curie temperature ($^\circ\text{C}$)
Iron (III) nitrate	404.00	47.2	858
Cobalt(II) nitrate	291.04	99.6	1121
Potassium hydroxide	56.11	406	240

The resultant precipitate was cleaned with pure water and kept at 70°C temperature for 8 hours. The as-prepared sample was annealed at 400°C - 600°C and 800°C for 6 hours. These nanoparticles were characterized by various techniques.

2.2. Experimental techniques.

The structural analysis was carried out through Philips analytical X-ray diffractometer ($\lambda=1.5401\text{\AA}$). The FT-IR spectrum taken through the PerkinElmer spectrometer ranges from 500 to $4,000\text{ cm}^{-1}$. The UV absorption studies were carried out through UV-2600, Shimadzu [11].

3. Results and Discussion

3.1. XRD analysis.

The XRD pattern of the investigated Co_2FeO_4 sample is shown in fig.1(a)–(d). The non-existence of peaks lines corresponds to the Co_2FeO_4 phase following heated samples at the various thermal condition. It has been observed that all the patterns split into two components for treated samples excluding at temperature 800°C . While comparing the peak position of all patterns, it evidence that the compound (Co_2FeO_4) is the spinel one.

The splitting of spectral lines is noted in Fig.1 (ranges from 20 to 80) and clearly shown for (311) XRD peak line alone in the right-hand side of Fig.1. The peak positions at higher and lower 2θ values are denoted at $2\theta_2$ and $2\theta_1$, respectively. The pattern for the untreated sample shown more splitting, and it is decreasing gradually closer and closer, and finally, it disappears at 800°C , suggesting a single phased compound.

There is a coexistence of two spinel phases confirmed from the lattice parameter a_1 and a_2 determined through $2\theta_1$ and $2\theta_2$, of the spectrum are presented in Table 2. The lattice parameter of the 800°C sample is a fair and good arrangement with theoretical value and experimental value.

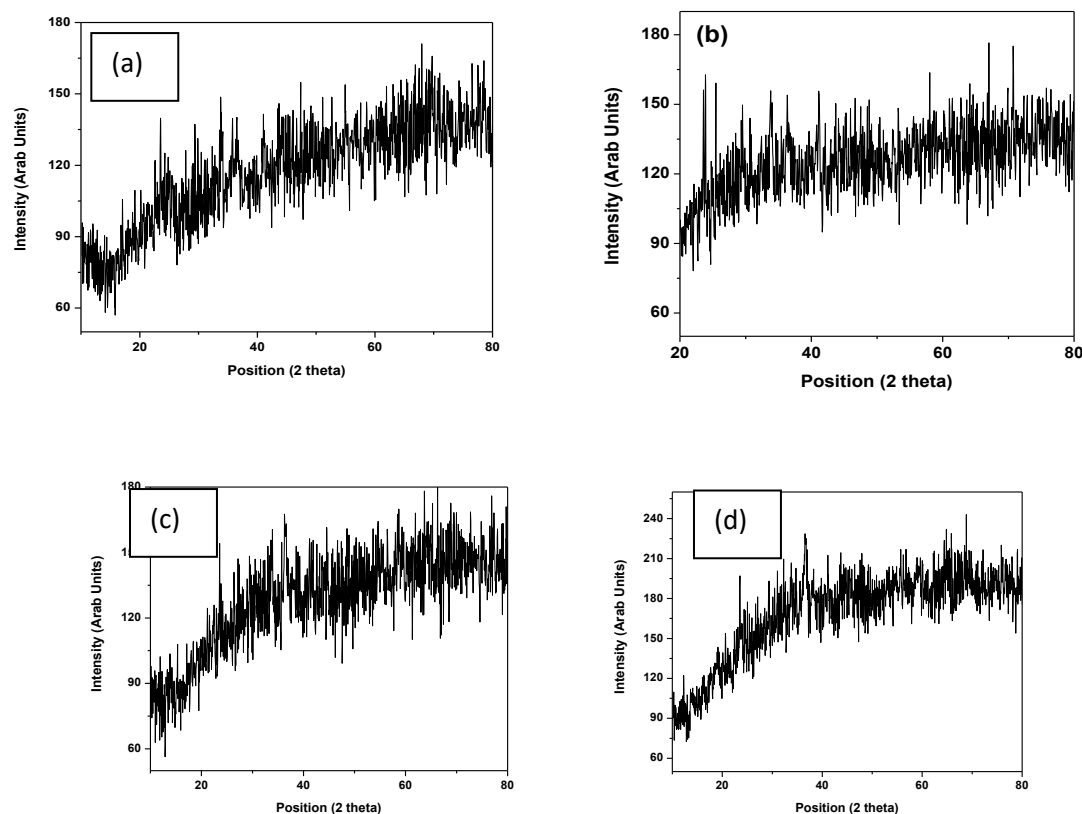


Figure 1. XRD spectra of at (a) room, (b) 400, (c) 600 and (d) 800 °C temperature.

It can be seen that the lattice parameter a_1 decreases as the temperature increases from 400°C to 800°C. Both (a_1 and a_2) are coinciding with 800°C (S90) sample.

Table 2. Crystallite size and lattice parameters.

Sample	$2\theta_1$	$2\theta_2$	Crystalline size (nm)	Lattice parameters a_1	Lattice parameters a_2
As-prepared	36.41	36.60	11.09	8.184	8.226
800°C	36.72	36.27	13.9	8.079	8.281

The particle size of the samples was calculated through well known Debye-Scherrer formula:

$$D = 0.89\lambda/\beta\cos\theta \quad (1)$$

($\lambda = 1.504 \text{ \AA}$), 2θ – peak position, D - particle size, β - FWHM) on (311) XRD peak at 2θ . The determined particles are in the nano range (~11 nm to 15nm) is due to various grain growth induced by the thermal energy of different annealing temperature [12, 13]. The crystalline size and lattice parameters of both as-papered and various annealed samples are as shown in the same Table 2.

3.2. FTIR investigation.

Figure 2 reveals that the FTIR spectra for the power sample heat traced at various temperatures. The FTIR spectrums of Co_2FeO_4 were recorded in the range 500-4000 cm^{-1} . The broad absorption peak at 3350 cm^{-1} , which corresponds to the O-H stretching vibration, arises from the molecules of the aqueous environment. The peak located at 2391 cm^{-1} is due to +ve atmosphere CO_2 present in the instrument stretching modes of C-C and C=O are observed of 1374 cm^{-1} , 1638 cm^{-1} , 1760 cm^{-1} , respectively. The band of 2391 cm^{-1} is ascribed to C-H

stretching vibration. The peak shift observed around 550cm^{-1} indication of the Co_2FeO_4 [14, 15]. Further, there is more absorption in annealed samples rather than as prepared.

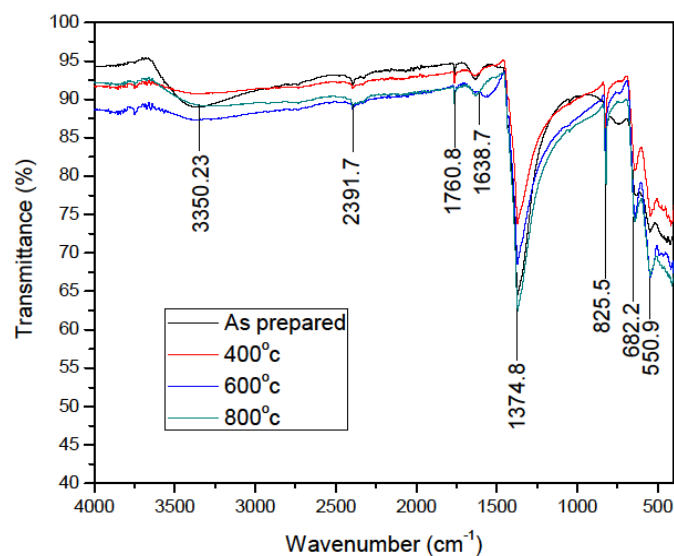


Figure 2. FTIR Spectra of Co_2FeO_4 at various temperatures.

3.3. UV-vis spectrometer(UV).

The UV-visible absorption has been used to monitor the optical properties of quantum sized particles. The fig 3 shows the absorption spectrum for Co_2FeO_4 nanoparticles of both as-prepared and annealed at temperatures 400°C , 600°C , and 800°C , respectively.

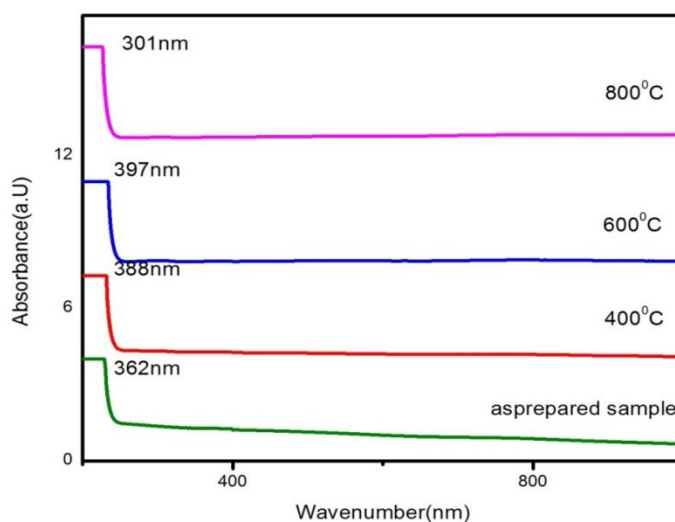


Figure 3. UV-vis absorbance spectra of Co_2FeO_4 .

The absorption spectra of both samples had a primary narrow peak and were found below 400nm . In addition to that, there are several numbers of shoulder peaks where observed in the visible range of the absorption spectrum. Absorption is depending on the temperature when increasing the temperature absorption gets decreased that is shown the same fig. 3. particle size increased absorbance decreased. The table shows the measurement from the UV-vis spectrometer.

Table 3. Absorbance and visible spectra.

Sample	Absorbance	Band gap (eV)
As prepared	362.9	3.41
400 ^o cm	388.8	3.19
600 ^o cm	397.5	3.12
800 ^o cm	301.0	4.12

3.4. Estimation of optical band gap.

Absorption in UV-vis spectral studies in the UV-Vis-NIR region was carried out to estimate the optical band gap (E_g) of the synthesized samples under study. These values are obtained through the well-known relations given by tauc and wood is given by,

$$\alpha h\nu = \beta(h\nu - E_g)^{n/2} \quad (2)$$

Where,

A - absorbance constant, h - plank constant and t- thickness of the cuvette and ν - frequency of UV source. The required E_g is the optical band gap, and n is the constant corresponds to different types of electronics transitions.

The calculated optical band gap of Co_2FeO_4 , both as-prepared and annealed samples of range (3 -4.2ev), are presented in Table 3. It is shown that when increasing the temperature, the optical band gap also increased due to the transformation of the phase structure—similar results were reported by some researchers [16,17].

4. Conclusions

From the above analysis, the following conclusions arrive, CoFe_2O_4 at as prepared and 400°C, 600°C, and 800°C were grown by the co-precipitation method. X-ray diffraction analysis substantiates the formation of a single-phase cubic spinel structure with nano-sized ferrites. The small change in lattice parameter with Co, may due to Co ions occupying Fe position in the lattice were also confirmed. The observed absorption bands divulge that the formation of spinel shape. Absorption measurements were performed with UV confirmed the optical bandgap of investigated samples.

Funding

This research received no external funding.

Acknowledgments

The authors are grateful to acknowledge the college authorities provided the necessary facility to complete this work.

Conflicts of Interest

The authors declare no conflict of interest.

References

1. Gabriel, L.; Mariella, A.; Cynthia, C.; Cabriel, B.; Jochen, L.F.; Elisa, B.S.; Internal structural and magnetic properties in Cobalt Ferrite Nanoparticle: Influence of the synthesis method. Particle and Particle system characterization, **2019**, 36, 1900061.

2. Bastami, T.R.; Entezari, M.H.; Hu, Q.H.; Hartono, S.B.; Qiao, S.Z. Role of polymeric surfactants on the growth of manganese ferrite nanoparticles. *Chemical Engineering Journal* **2012**, *210*, 157-165, <https://doi.org/10.1016/j.cej.2012.08.031>.
3. Supriya, S.; Kumar, S.; Kar, M. Correlation between AC and DC transport properties of Mn substituted cobalt ferrite. *Journal of Applied Physics* **2016**, *120*, <https://doi.org/10.1063/1.4968795>.
4. Sivakumar, M.; Kanagesan, S.; Umapathy, V.; Chinnaraj, K.; Nithiyantham, S. Study of CoFe₂O₄ Particles Synthesized with Various Concentrations of PVP Polymer. *Journal of Superconductivity and Novel Magnetism* **2013**, *26*, 725-731.
5. Sivakumar, M.; Kanagesan, S.; Chinnaraj, K.; Suresh Babu, R.; Nithiyantham, S. Synthesis, Characterization and Effects of Citric Acid and PVA on Magnetic Properties of CoFe₂O₄. *Journal of Inorganic and Organometallic Polymers and Materials* **2013**, *23*, 439-445, <https://doi.org/10.1007/s10904-012-9801-x>.
6. Sumanth, Y.A.; Sujatha, R.A.; Mahalakshmi, S.; Karthika, P.; Nithiyantham, S.; Saravanan, S.; Azagiri, . Synthesis and characterization of nanophase zinc oxide materials. *Journal of Material Science – Materials in Electronics*, **2016**, *27*, 1616-1621.
7. Hayrapetyan, S.S.; Khachatryan, H.G. Porosity appearance in cobalt ferrite and changes under the thermal treatment of co-precipitated iron(II) and cobalt(II) oxalates. *Microporous and Mesoporous Materials* **2004**, *72*, 105-110, <https://doi.org/10.1016/j.micromeso.2004.04.023>
8. Nikumbh, A.K.; Pawar, R.A.; Nighot, D.V.; Gugale, G.S.; Sangale, M.D.; Khanvilkar, M.B.; Nagawade, A.V. Structural, electrical, magnetic and dielectric properties of rare-earth substituted cobalt ferrites nanoparticles synthesized by the co-precipitation method. *Journal of Magnetism and Magnetic Materials* **2014**, *355*, 201-209, <https://doi.org/10.1016/j.jmmm.2013.11.052>.
9. Jiao, Z.; Geng, X.; Wu, M.; Jiang, Y.; Zhao, B. Preparation of CoFe₂O₄ nanoparticles by spraying co-precipitation and structure characterization. *Colloids Surface Science – A* **2008**, *313*, 31-34, <https://doi.org/10.1016/j.colsurfa.2007.04.066>.
10. Kiran, V.S.; Sumathi, S. Comparison of catalytic activity of bismuth substituted cobalt ferrite nanoparticles synthesized by combustion and co-precipitation method. *Journal of Magnetism and Magnetic Materials* **2017**, *421*, 113-119, <https://doi.org/10.1016/j.jmmm.2016.07.068>.
11. . Mahalakshmi, S.; Jayasri, R.; Nithiyantham, S.; Swetha, S.; Santhi, K, Magnetic interactions and dielectric behavior of cobalt ferrite and barrium titanate multiferroics nanocomposites, *Applied Surface Science*, **2019**, *494*, 51-56.
12. Mustaq, M.W.; Imran, M.; Bashir, S.; Kanwal, F.; Mitu, L. Synthesis, structural and biological studies of cobalt ferrite nanoparticles. *Bulgarian Chemical Communications* **2016**, *48*, 565.
13. Rahman, O.u.; Mohapatra, S.C.; Ahmad, S. Fe₃O₄ inverse spinal super paramagnetic nanoparticles. *Materials Chemistry and Physics* **2012**, *132*, 196-202, <https://doi.org/10.1016/j.matchemphys.2011.11.032>.
14. Viviliya, S. Synthesis the characterization of cobalt ferrite by co-precipitation method, MSc, Thesis, submitted to Bharathidasan University, Tamilnadu, India, **2017**.
15. Raut, A.; Barkule, R.; Shengule, D.; Jadhav, K.J. M. Synthesis, structural investigation and Magnetic Properties of Zn ²⁺ Substituted Cobalt Ferrite Nanoparticles Prepared by the Sol–Gel Auto-Combustion Technique. *Journal of magnetism and magnetic materials*, **2014**, *359*, 87-92.
16. Kalam, A.; Al-Sehemi, A.G.; Assiri, M.; Du, G.; Ahmad, T.; Ahmad, I.; Pannipara, M. Modified solvothermal synthesis of cobalt ferrite (CoFe₂O₄) magnetic nanoparticles photocatalysts for degradation of methylene blue with H₂O₂/visible light. *Results in Physics* **2018**, *8*, 1046-1053, <https://doi.org/10.1016/j.rinp.2018.01.045>.
17. Meenal, G.; Anushree, D.; Satyabratha, M.; Dipankar, D.; Anindya, D. Surfactant based synthesis and magnetic studies of cobalt ferrite, *Applied Physics, A*, **2020**, *126*, 660. <https://doi.org/10.1007/s00339-020-03823-9>.