



UNIVERSITI PUTRA MALAYSIA

***STUDY ON THE PROPERTIES OF COBALT FERRITE NANOPARTICLES
PREPARED BY CO-PRECIPITATION AND COBALT FERRITE/CARBON
BLACK NANOCOMPOSITES***

NUR ATIQA BINTI ADNAN

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BLACK NANOCOMPOSITES**

By

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**Thesis Submitted to the Department of Physics, Universiti Putra Malaysia, in
partial Fulfilment of the Requirements for the Degree of Bachelor of Science
with Education (Honours) Major in Physics**

FEBRUARY 2022

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DEDICATION

I dedicated this work to every person that has encouraged and helped me throughout the completion of this research.



ABSTRACT

STUDY ON THE PROPERTIES OF COBALT FERRITE NANOPARTICLES PREPARED BY CO-PRECIPITATION AND COBALT FERRITE/CARBON BLACK NANOCOMPOSITES

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FEBRUARY 2022

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Cobalt ferrite nanoparticles with the chemical formula of CoFe_2O_4 and their nanocomposites with carbon black were synthesized by the previous final year project student through the co-precipitation method. Cobalt ferrite nanoparticles, CoF, were sintered at a temperature of 800°C in a carbolite tube furnace for 4 hours. The synthesized cobalt ferrite nanoparticles were mixed with carbon black, CB with different weight percentages of carbon black, such as 2%, 4%, 6%, 8%, and 10% to produce cobalt ferrite/carbon black, CoFCB nanocomposites. The characterization of cobalt ferrite, carbon black, and cobalt ferrite/carbon black nanocomposites for the structural, morphological, optical, magnetic, and electrical properties of the nanocomposites were done by using X-Ray Diffraction (XRD), Field Emission

Scanning Electron Microscopy (FESEM), Ultraviolet Visible Spectroscopy (Uv-Vis), 2-point probe, and Vibrating Sample Magnetometer (VSM). Cobalt ferrite nanoparticles were successfully synthesized as the XRD pattern of CoF shows peaks of single-phase spinel cubic structure of ferrite. There was no secondary phase visible in the XRD analysis of the CoFCB nanocomposite. The particle size distribution of CoF nanoparticles ranges from 50-110 nm with an average particle size of 73 nm. For optical properties, the optical band gap energy of CoF is 1.36 eV while the optical band gap energy of CB is 1.01 eV. With increasing carbon black concentration, the band gap energy of CoFCB nanocomposites decreased from 1.24 eV to 1.04 eV. For electrical properties, the resistivity of CoF is $6.21 \times 10^6 \Omega\text{m}$ and CB is $9.31 \times 10^2 \Omega\text{m}$. For conductivity, the conductivity of CB is $1.07 \times 10^{-3} \Omega\text{m}$ while the conductivity of CoF is $1.61 \times 10^{-8} \Omega\text{m}$. As the content of carbon black in CoFCB nanocomposite increased, the resistivity decreased while the conductivity increased. The saturation magnetization for CoF was found to be 89.768 emu/g while the saturation magnetization for CB was 2.0797 emu/g. The saturation magnetization value for the CoFCB2% to CoFCB10% nanocomposite ranged between 73.083 emu/g and 102.65 emu/g. For the retentivity of nanocomposites, the value ranged from 37.56 emu/g to 52.229 emu/g. The values of coercivity of nanocomposites varied from 944.83 G to 1179.7 G.

ABSTRAK

KAJIAN MENGENAI SIFAT-SIFAT NANOZARAH FERIT KOBALT YANG DISEDIAKAN MELALUI PEMENDAKAN BERSAMA DAN NANOKOMPOSIT FERIT KOBALT/KARBON HITAM

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Nanozarah ferit kobalt dengan formula kimia CoFe_2O_4 dan nanokompositnya dengan peratusan berat karbon hitam yang berbeza telah disintesis oleh pelajar tahun akhir projek sebelum ini dengan menggunakan kaedah pemendapan bersama. Nanopartikel ferit kobalt, CoF telah disinter pada suhu 800°C dalam relau tiub karbolit selama 4 jam. Nanozarah ferit kobalt yang telah disintesis dicampur dengan karbon hitam, CB dengan peratusan berat karbon hitam yang berbeza iaitu 2%, 4%, 6%, 8%, dan 10 % untuk menghasilkan nanokomposit ferit kobalt/karbon hitam, CoFCB. Sifat-sifat ferit kobalt, karbon hitam dan nanokomposit ferit kobalt/karbon hitam iaitu sifat struktur, morfologi, optik, magnetik dan elektrik bagi nanokomposit telah dilakukan dengan

menggunakan Pembelauan Sinar-X (XRD), Mikroskop Elektron Imbasan Pancaran Medan (FESEM), Spektroskopi Ultraungu-Nampak (Uv-Vis), Prob Dua Titik dan Magnetometer Sampel Bergetar (VSM). Nanopartikel ferit kobalt telah berjaya disintesis kerana corak XRD CoF menunjukkan puncak-puncak struktur kubik spinel fasa tunggal untuk ferit. Dalam analisis XRD, nanokomposit CoFCB, tidak ada penampakan fasa sekunder. Taburan saiz zarah nanozarah CoF adalah di antara 50-110 nm dengan saiz zarah purata 73 nm. Untuk sifat optik, tenaga jurang jalur optik CoF ialah 1.36 eV manakala tenaga jurang jalur optik CB ialah 1.01 eV. Tenaga jurang jalur berkurangan daripada 1.24 eV kepada 1.04 eV dengan peningkatan kandungan karbon hitam dalam nanokomposit ferit kobalt/karbon hitam. Untuk sifat elektrik, kerintangan CoF ialah $6.21 \times 10^6 \Omega \cdot m$ dan CB ialah $9.31 \times 10^2 \Omega \cdot m$. Bagi kekonduksian, kekonduksian CB ialah $1.07 \times 10^{-3} \Omega \cdot m$ manakala kekonduksian CoF iaitu $1.61 \times 10^{-8} \Omega \cdot m$. Apabila kandungan karbon hitam dalam nanokomposit CoFCB meningkat, kerintangan berkurangan manakala kekonduksian meningkat. Pemagnetan tepu untuk CoF ialah 89.768 emu/g manakala pemagnetan tepu CB ialah 2.0797 emu/g. Untuk nanokomposit CoFCB2% hingga CoFCB10%, nilai pemagnetan tepu adalah di antara 73.083 emu/g hingga 102.65 emu/g. Nilai pemagnetan baki bagi nanokomposit adalah di antara 37.56 emu/g hingga 52.229 emu/g. Nilai koersiviti bagi nanokomposit adalah di antara 944.83 G hingga 1179.7 G.

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TABLE OF CONTENTS

	Page
DEDICATION	i
ABSTRACT	ii
<i>ABSTRAK</i>	iv
ACKNOWLEDGEMENTS	vi
APPROVAL	vii
DECLARATION	viii
LIST OF FIGURES	xii
LIST OF TABLES	xiv
LIST OF ABBREVIATIONS	xv
CHAPTER	
1 INTRODUCTION	
1.1 Ferrite	1
1.2 Cobalt Ferrite	2
1.3 Carbon Black	3
1.4 Problem Statement	4
1.5 Objectives	4
2 LITERATURE REVIEW	
2.1 Review of Past Research	6
2.1.1 Cobalt Ferrite and Carbon-based Material Nanocomposites	6
2.2 Magnetism	10
2.2.1 Diamagnetism	11

2.2.2	Paramagnetism	11
2.2.3	Ferromagnetism	12
2.2.4	Antiferromagnetism	13
2.2.5	Ferrimagnetism	13
2.3	Hysteresis Loop	14
2.4	Structure of Spinel Ferrite	16
2.5	Cobalt Ferrite	17
2.6	Co-precipitation Method	18
2.7	Composite	19
2.8	Optical Band	20
2.9	Electrical Properties	21
3	METHODOLOGY	
3.1	Introduction	23
3.2	Experimental Procedure	25
3.2.1	Raw Materials	25
3.2.2	Weighing	25
3.2.3	Mixing	26
3.2.4	Filtering and Rinsing Process	27
3.2.5	Drying	28
3.2.6	Grinding	28
3.2.7	Sintering	28
3.2.8	Composite Preparation	30
3.3	Characterization	31
3.3.1	X-Ray Diffraction (XRD)	31

3.3.2	Field Emission Scanning Electron Microscopy (FESEM)	33
3.3.3	Vibrating-Sample Magnetometer (VSM)	34
3.3.4	Ultraviolet-visible (UV-Vis) Spectroscopy	35
3.3.5	Two-Point Probe	36
4	RESULTS AND DISCUSSION	
4.1	Introduction	38
4.1	XRD Analysis	38
4.2	FESEM Analysis	41
4.3	Optical Band Gap	47
4.4	Electrical Properties	50
4.5	Magnetic Properties	51
5	CONCLUSION AND SUGGESTION	
5.1	Conclusion	56
5.2	Suggestion	58
	REFERENCES	59
	APPENDICES	63
	VITAE	78

LIST OF FIGURES

Figure		Page
2.1	Magnetic moment of diamagnetism when there are absence and presence of external magnetic field	11
2.2	Magnetic moment of paramagnetism when there are absence and presence of external magnetic field	12
2.3	Magnetic moment of ferromagnetism when there are absence and presence of external magnetic field	12
2.4	Magnetic moment of antiferromagnetism when there are absence and presence of external magnetic field	13
2.5	Magnetic moment of ferrimagnetism when there are absence and presence of external magnetic field	14
2.6	Hysteresis loop	16
2.7	Spinel ferrite structure	17
2.8	Band diagrams for a conductor, semiconductor, and insulator	21
3.1	Flowchart of sample preparation	24
3.2	Electronic analytical balance	26
3.3	Hotplate and magnetic stirrer	27
3.4	Filtration process	27
3.5	Pestle and mortar	28
3.6	Carbolite tube furnace	29
3.7	Graph of sintering time to prepare cobalt ferrite nanoparticle	29
3.8	X-Ray Diffraction Instrument	32
3.9	Field Emission Scanning Electron Microscopy (FESEM)	33
3.10	Vibrating sample magnetometer (VSM)	34

3.11	Schematic diagram of UV-Vis setup	35
3.12	Schematic diagram of Two-point probe measurement	36
4.1	XRD Pattern of CoF, CB and CoFCB nanocomposites with varying weight	39
4.2	FESEM Micrographs of CoF and CB	42
4.3	Particle Size Distribution Histogram of CoF and CB	43
4.4	FESEM micrograph of CoFCB nanocomposite at 100k magnification	45
4.5	EDX Spectra of CoF, CB and CoFCB with varying weight percentage of carbon black content	46
4.6	Graph of absorbance versus wavelength of CoF, CB and CoFCB nanocomposites	47
4.7	Graph of reflectance versus wavelength of CoF, CB and CoFCB nanocomposites	48
4.8	Magnetic hysteresis loops for (a) CoF and CoFCB nanocomposites (b) CB	53

LIST OF TABLES

Tables	Page
3.1 Weight percentage and mass of cobalt ferrite and carbon black in each sample	30
4.1 Optical band gap energy of CoF, CB and CoFCB nanocomposites	50
4.2 Resistivity and Conductivity Values of CoF, CB and CoFCB nanocomposites	51
4.3 Saturation Magnetization, Retentivity and Coercivity Values of cobalt ferrite, carbon black, and cobalt ferrite/carbon black Nanocomposites	55

LIST OF ABBREVIATIONS

CoF	Sample of cobalt ferrite
CoFCB2%	Sample of cobalt ferrite/carbon black nanocomposite with carbon black weight percentage of 2%
CoFCB4%	Sample of cobalt ferrite/carbon black nanocomposite with carbon black weight percentage of 4%
CoFCB6%	Sample of cobalt ferrite/carbon black nanocomposite with carbon black weight percentage of 6%
CoFCB8%	Sample of cobalt ferrite/carbon black nanocomposite with carbon black weight percentage of 8%
CoFCB10%	Sample of cobalt ferrite/carbon black nanocomposite with carbon black weight percentage of 10%
CB	Sample of carbon black
XRD	X-Ray Diffraction
FESEM	Field Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-Ray Analysis
UV-VIS	Ultraviolet Visible Spectroscopy
VSM	Vibrating Sample Magnetometer
wt%	Weight percentage
Co(NO ₃) ₂	Cobalt Nitrate salt
Fe(NO ₃) ₃	Ferric Nitrate salt
Å	Angstrom (10 ⁻¹⁰ m)

CHAPTER 1

INTRODUCTION

1.1 Ferrite

A ferrimagnetic material or ferrite is a magnetic material whose atomic moments are aligned in an unequal or opposite direction. Ferrite is a hard and brittle ceramic-like polycrystalline material usually has gray or black color. The typical formula of ferrite is $M^{2+}Fe_2^{3+}O_4^{2-}$ which is equivalent to $MOFe_2O_3$, where M^{2+} is a dispositive metal ion such as Fe^{2+} , Co^{2+} , and others. Ferrite can be classified by its crystal structural properties: spinel ferrite, garnet ferrite, orthoferrite, and hexagonal ferrites. Besides that, ferrites are also classified based on their magnetic properties, which as hard ferrite and soft ferrite.

Ferrites have unique electrical and magnetic characteristics useful for many applications due to their high magnetic permeability, which contributes to spontaneously getting magnetized, low electrical conductivity, which means high electrical resistance and minimum loss of eddy current. Permanent ferrite magnets are constructed of hard ferrites with strong coercivity and remanence after magnetization, therefore difficult to be demagnetized. Ferrite has high magnetic permeability and conducts magnetic flux well, allowing them to store magnetic fields that are more powerful than iron (Jadhav et al., 2020). Ferrite also has high electrical resistance and is not electrically conductive, making them suitable for applications such as magnetic cores for transformers to control eddy currents (Spaldin et al., 2012).

There are a few significant applications of ferrite in business and everyday life, such as ac and dc motors, power transformers, video and audio applications, microwave devices, data storage systems, permanent magnets, and others (Valenzuela,

2005). Therefore, the synthesis method should be chosen wisely based on ferrite products' desired structural and morphologies (Jadhav et al., 2020). Some synthesis methods for ferrite include thermal synthesis, sol-gel, solid-state reactions, and co-precipitation. Synthesis methods significantly affect the size and surface area control of ferrite products.

1.2 Cobalt Ferrite

Cobalt is the sole element that increases saturation magnetization and curie temperature when alloyed with iron. As a result, cobalt iron alloys are gaining interest in the scientific community. Cobalt ferrite is sought after for many applications due to its strong anisotropy, high coercivity, moderate saturation magnetization, good mechanical stability, and high chemical stability at higher temperatures. Cobalt ferrite is suited for recording applications such as audio and videotapes and digital recording discs, and new research has revealed that they exhibit photomagnetic characteristics (Chinnasamy et al., 2003). Cobalt ferrite is an excellent resource for various applications, including biomedicine in human pancreatic and ovarian cancer cells, alkene oxidation catalysts, adsorbents for effective removal of arsenic, methanol decomposition catalysts, and microwave absorption media (Fitriyanti et al., 2017). The wide range of applications for nano ferrites, including permanent magnets, ferrofluids, storage devices, and other applications, has sparked much interest in how these nanomaterials are synthesized. (Houshiar et al., 2014).

Cobalt ferrite nanoparticle is synthesized by several conventional methods, including combustion methods, electrochemical synthesis, sol-gel method, hydrothermal synthesis, citrate precursor techniques, solid-state reaction, and co-

precipitation. The crystal structure, size, morphology, and distribution of cobalt ferrite will be affected by suitable synthesis methods and conditions, which will impact the magnetic characteristics of cobalt ferrite (Chinnasamy et al., 2002). Therefore the synthesis method of cobalt ferrite nanoparticles is essential as it will affect the properties of the nanoparticles.

1.3 Carbon Black

Carbon black is a paracrystalline carbon mostly composed of pure carbon, but other elements might be present in carbon black, such as oxygen, hydrogen, nitrogen, and sulphur. The shape of carbon black particles is typically spherical and has irregular crystallinity. Carbon black is primarily used in the application of energy storage and commercial products such as carbon brushes, UV absorbers, automotive tires, electrodes, printing inks, and others. Carbon black has low density, large specific surface area, outstanding electrical conductivity, excellent chemical and mechanical stability. Carbon black is also inexpensive and readily available, which helps lower the cost of device production. Carbon compounds such as carbon fibers, carbon black, and graphite are commonly utilized as fillers in composite materials that offer electromagnetic shielding without the need for highly conductive metal elements. Carbon compounds like carbon black are common electromagnetic absorbers with large permittivity values owing to its high electric conductive loss and relaxation loss.

1.4 Problem Statement

Nowadays, ferrite is one of the most sought-after materials in high-frequency applications due to its large resistivity, low conductive losses, and high permeability (Dabagh, 2016). Many researchers are interested in cobalt ferrite due to the inverse spinel structure, which contributes to many of their magnetic and electrical properties. To support the potential of the latest application, the synthesis process and material modification and any characterizations of the ferrite nanoparticles' structure and other characteristics should be carried out. Furthermore, cost reduction during the synthesis of ferrite nanoparticles has become one of the essential functions to support the latest application. Therefore, co-precipitation was used as the preferred method of synthesizing cobalt ferrite/carbon black nanocomposites because of its simplicity, low cost, and reasonable size control. Carbon black is used to change the characterizations and properties of cobalt ferrite to investigate the properties of using cobalt ferrite/carbon black nanocomposites. This study will discuss and analyze the secondary data which is the data that is collected by the final year project student to investigate the properties of cobalt ferrite/carbon black nanocomposite by combining cobalt ferrite and carbon black by varying the percentages of the content of the carbon black according to its weight percentage to form a nanocomposite.

1.5 Objectives

The objectives of this research are:

1. To discuss the methodology of synthesizing cobalt ferrite nanoparticles by using the co-precipitation method and the synthesization of cobalt

ferrite/carbon black nanocomposites by varying the content of carbon black at weight percentages of 2%, 4%, 6%, 8%, and 10%.

2. To process secondary data of cobalt ferrite, carbon black, and cobalt ferrite/carbon black nanocomposites characterized by X-Ray diffraction (XRD), Field Emission Scanning Electron Microscope (FESEM), 2 point-probe, Ultraviolet-visible (UV-Vis) Spectrometer, and Vibrating Sample Magnetometer (VSM) respectively.
3. To analyze the structural, morphological, optical band gap, electrical and magnetic properties of cobalt ferrite, carbon black, and cobalt ferrite/carbon black nanocomposites.

CHAPTER 2

LITERATURE REVIEW

2.1 Review of Past Research

This section will discuss past research related to cobalt ferrite nanocomposites with various carbon materials such as graphene, multi-walled carbon nanotubes, carbon black, and others.

2.1.1 Cobalt Ferrite and Carbon-based Material Nanocomposites

Xia et al. (2012) researched synthesizing cobalt ferrite nanoparticles using the hydrothermal method and characterizing cobalt ferrite/graphene nanocomposite. The content of graphene in cobalt ferrite/graphene nanocomposite was varied in weight percentage (0, 10, 20, and 30 wt%). XRD pattern revealed no diffraction peaks of GO (001) at the patterns of samples with graphene content of 10% and 20%, which may be due to reduction of GO to graphene due to usage of alcohol in the hydrothermal reaction. The result showed weak diffraction peaks for graphene in both samples due to the graphene layer destroying the usual stacking of layer or randomly restacking by the reduction process. The morphologies of pure cobalt ferrite had high aggregation. The research revealed that when the content of graphene in the samples increased, the aggregation was shown to decrease due to their large specific surface area. At the sample with graphene content of 20 wt%, the graphene nanosheets had a small amount of aggregation. Cobalt ferrite/graphene nanocomposite with 20 wt% of graphene content had high reversible specific heat, good rate capability, and excellent cycling stability.

Bakhshi et al. (2016) had published a paper about the synthesis and the effects of coating time on the characterization of cobalt ferrite coated with carbon shell

nanoparticles. The cobalt ferrite nanoparticles were synthesized using thermal chemical vapor deposition (TVCD). The coating time of nanocomposites was varied for 1, 2, and 3 hours. The XRD patterns indicated that the carbon-coated cobalt ferrite nanoparticles showed a graphite structure. When the XRD patterns and Tama spectra were compared, the results of all samples were amorphous. The simultaneous thermal analysis showed weight loss for the increasing temperature of 50 to 610°C, but no weight loss was observed as the temperature increased greater than 620°C. From the image of TEM and HRTEM, there were well-formed carbon shells on the surface of the samples. There was also the presence of ordered carbon layers (graphite) and amorphous carbon. From the VSM result, the diamagnetic graphite reduced the magnetic saturation and coercivity of the nanocomposites sample. The research showed that as the coating temperature increased, the growth of cobalt ferrite nanoparticles increased, which influenced the magnetic saturation. When the grain size decreased until the domain size, the coercivity increased, but if the grain size further decreased below the domain size, the coercivity decreased due to the rotation of the domain. The results from the micro-Raman spectra showed that the additional carbon was deposited onto the core of the sample as the coating time increased.

A study about maximizing the optimization of the physical properties of cobalt ferrite/graphene nanocomposites was conducted by Ateia et al. (2019). Cobalt ferrite nanoparticles, CoFe_2O_4 and cobalt ferrite/graphene nanocomposites, $\text{CoFe}_2\text{O}_4/\text{GR}$ were synthesized using the citrate auto-combustion method. In this study, they investigate how the content of graphene influence the structural, magnetic, and optical properties of pure cobalt ferrite and cobalt ferrite/graphene nanocomposites with weight percentages of 0.01 and 0.02 wt%. The XRD patterns of the sample showed the formation of the single-phase cubic spinel structure. The diffraction peak of the

nanocomposites became broader as the graphene concentration increased due to the average particle diameter decreasing. The images from the scanning electron microscopy showed that the CoFe_2O_4 sample grain shape is rough and regular, and the agglomeration is high while CoFe_2O_4 nanoparticles in $\text{CoFe}_2\text{O}_4/\text{GR}$ nanocomposite are regularly absorbed on the graphene sheets. The band gap energy increased as the graphene content increased due to the sample's small crystallite size and change in the electronic structure and charge density. The magnetization hysteresis loop of $\text{CoFe}_2\text{O}_4/\text{GR}$ showed the same ferromagnetic material trend as the pure CoFe_2O_4 sample. The saturation magnetization of the nanocomposites decreased while the coercivity increased as the content of graphene decreased. The calculated anisotropy constant showed that the anisotropy decreases as the graphene content in the nanocomposites increased.

Mubasher & Mumtaz (2021) researched the synthesis, structural, dielectric, and impedance properties of cobalt ferrite/multi-walled carbon nanotubes nanocomposites $\text{CoFe}_2\text{O}_4/(\text{MWCNTs})_x$. Cobalt ferrite nanoparticles were synthesized using co-precipitation, and their nanocomposites were produced using the ultrasonication method. Nanocomposites' characterization was analyzed using XRD, scanning electron microscopy (SEM), and LCR meter. The weight percentages of the MWCNT in the $\text{CoF}/(\text{MWCNTs})$ are 0, 10, 20, and 30 wt%. The XRD patterns showed the face-centered cubic structure of cobalt ferrite nanoparticles. The density of nanocomposites decreased as the concentration of MWCNTs increased as the porous volume increased because of the porous nature of MWCNTs. In comparison, the specific surface area increases because the surface-to-volume ratio of MWCNTs is high. The dielectric parameters of the nanocomposites are better than those of the pure cobalt ferrite nanoparticles. As the concentration of MWCNT in the nanocomposites

increased, the dielectric parameters also increased due to the assistance of MWCNT in the conduction mechanism. Besides, the improvement in the conduction mechanism caused the conductivity of $\text{CoF}/(\text{MWCNTs})_x$ nanocomposites to increase, and the resistance decreased as the inclusion of MWCNT increased. The impedance value of the sample with the highest concentration of MWCNT was the lowest, which confirms the potential of $\text{CoF}/(\text{MWCNTs})_x$ nanocomposites usage as high-performance energy storage materials.

Zhang et al. (2021) studied the preparation of carbon nanofiber-supported cobalt ferrite composites (C/CoFO) by electrospinning and hydrothermal methods. They also analyzed the composites' morphology, structure, and microwave absorption characteristic with weight percent of cobalt ferrite varies of 5, 10, 15, and 100%. The SEM results showed that the pure CoFO nanoparticles have medium agglomeration while the pure C nanofibers have continuous smooth surfaces. After the inclusion of CoFO, the composites still have a fibrous structure, and there are CoFO nanoparticles observed on the surface of C nanofibers. CoFO nanoparticles in the composites have uniform particle distribution and smaller grain sizes than the pure CoFO sample. The XRD pattern of pure CoFO nanoparticles showed the characteristic diffraction peak of the spinel structure of cobalt ferrite. Compared to the pure CoFO nanoparticles, the C/CoFO composites have smaller peaks, and the XRD patterns are broader due to the smaller particle size of CoFO nanoparticles. The hysteresis loop of pure CoFO nanoparticles showed large saturation magnetization and small coercive force. The saturation magnetization of C/CoFO composites is lower than pure CoFO nanoparticles. The composites have larger coercivity after the inclusion of CoFO nanoparticles. The electromagnetic properties were measured by using VNA. As the concentration of CoFO nanoparticles increased, the permeability values increased, but

then the value decreased. The permeability values when the content of CoFO nanoparticles was less than 10 wt% was larger than pure C nanofibers, but when the content of CoFO nanoparticles increased to 15 wt%, the dielectric parameters have rapidly decreased. From this research, as the CoFO nanoparticles content in the sample increases, the impedance, and magnetic loss ability increase. The best impedance value is when the content of CoFO nanoparticles was 10 wt%.

2.2 Magnetism

Magnetism is a phenomenon where magnetic fields cause the motion of electric charges. There are motions of electrons that contribute to the magnetic behavior of material: spin motion, where the motion of an electron spinning about its axis, and the orbital motion of the electron around the atomic nucleus. These two motions cause the difference in magnetic moments, which contribute to magnetism.

Two effects cancelled the magnetic moments related to the electron spin and orbital motion, making most materials not display magnetism. One of the effects is related to the Pauli exclusion principle, which states that two electrons in the same orbit will have opposing spins, resulting in distinct magnetic moments. Because all electron spin-orbital moments in a filled shell cancel, most elements' atoms have no net magnetic moment. All materials are affected by magnetic fields, but each material responds to the magnetic field differently. These behaviors will affect the strength of magnetization of materials. The materials display several types of behavior when they are exposed to a magnetic field, such as diamagnetism, paramagnetism, ferromagnetism, antiferromagnetism and ferrimagnetism.

2.2.1 Diamagnetism

In diamagnetic materials, the magnetization of the materials is less than zero because the direction magnetization is opposite to the direction of the applied magnetic field. The atomic magnetic dipole was produced by the magnetic field, which has an effect on the magnetic moment caused by rotating electrons. The magnetic field is repelled by these dipoles, resulting in a magnetization of less than zero. A diamagnetic substance has a negative susceptibility or a relative permeability of around 1. Examples of materials displaying diamagnetism at room temperature are copper, silver, and gold. Figure 2.1 shows the magnetic moments of diamagnetism when there is an external magnetic field's absence and presence.

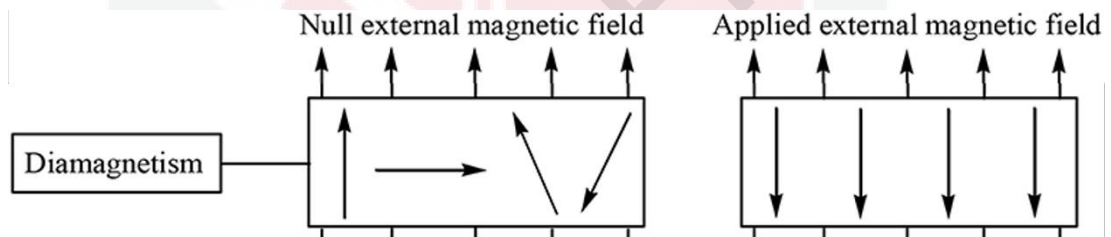


Figure 2.1: Magnetic moment of diamagnetism when there are absence and presence of external magnetic field (*Dia-magnetism*, n.d.)

2.2.2 Paramagnetism

When materials contain unpaired electrons, each atom has a net magnetic moment due to electron spin. When a magnetic field is applied, the dipoles align with the field, resulting in positive magnetization. Due to the lack of interaction between dipoles, large magnetic fields are required to align all of them. When the magnetic field is withdrawn, the effect is gone. Paramagnetism is a phenomenon that occurs in metals such as aluminum, titanium, and copper alloys. Above the Curie temperature, ferromagnetic and ferrimagnetic materials display paramagnetism. Figure 2.2 shows

the magnetic moment of paramagnetism when there is an external magnetic field's absence and presence.

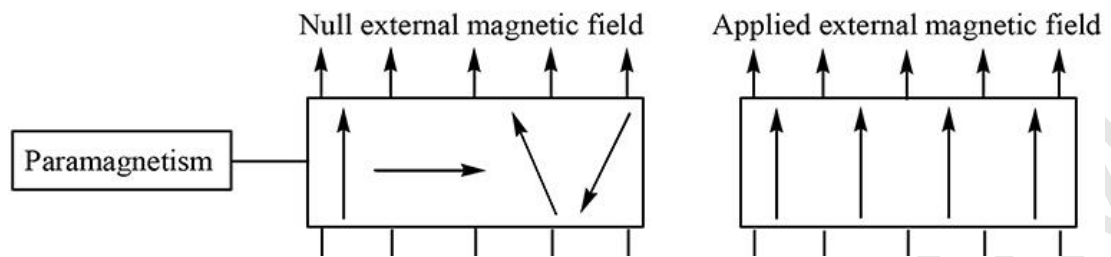


Figure 2.2: Magnetic moment of paramagnetism when there are absence and presence of external magnetic field (*Dia-magnetism*, n.d.)

2.2.3 Ferromagnetism

The unfilled energy levels in the 3d orbital level in metals such as iron, nickel, and cobalt induce ferromagnetic behavior. Due to the dipoles' exchange interaction, permanent unpaired dipoles in ferromagnetic materials soon line up with the applied magnetic field. Even for minimal magnetic fields, huge magnetizations may be generated, resulting in enormous susceptibilities approaching 10^6 . The susceptibility of ferromagnetic materials is determined by the strength of the applied magnetic field. Ferromagnetic materials act as paramagnetic materials at a temperature higher than the Curie temperature. Figure 2.3 shows the magnetic moment of diamagnetism when there is an external magnetic field's absence and presence.

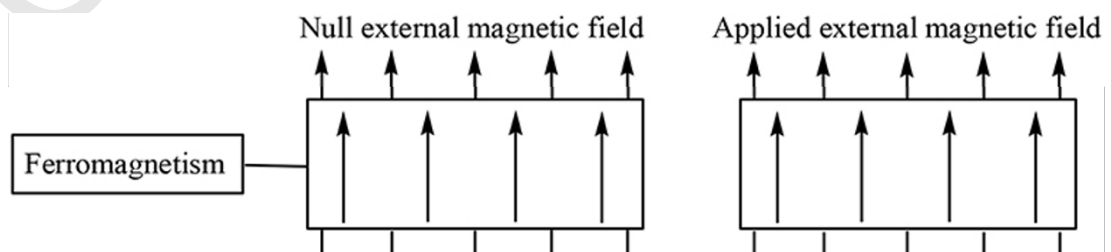


Figure 2.3: Magnetic moment of ferromagnetism when there are absence and presence of external magnetic field (*Dia-magnetism*, n.d.)

2.2.4 Antiferromagnetism

Antiferromagnetic materials like manganese and chromium have no magnetism. Even though the magnitude of each dipole is really great, the magnetic moments induced in surrounding dipoles line up in the opposite direction from one another in the magnetic field. Magnetic susceptibility is weak and positive. Figure 2.4 shows the magnetic moment of antiferromagnetism when there is an external magnetic field's absence and presence.

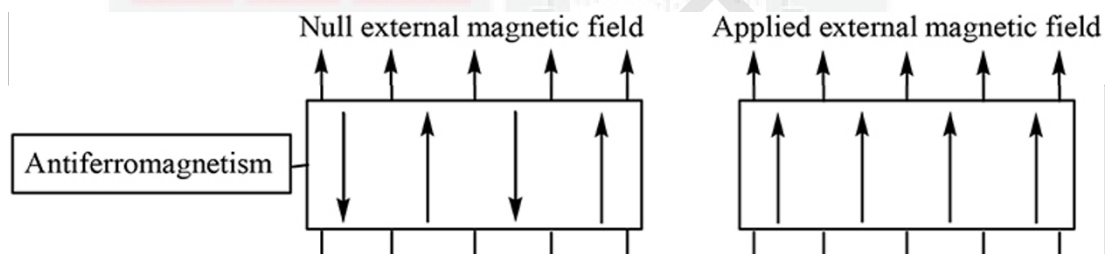


Figure 2.4: Magnetic moment of antiferromagnetism when there are absence and presence of external magnetic field (*Dia-magnetism*, n.d.)

2.2.5 Ferrimagnetism

Different ions have different magnitudes of magnetic moments in ceramic materials. Because the intensity or quantity of dipoles is not equal, the dipoles of cation A may line up with the field while the dipoles of cation B oppose the field in a magnetic field, resulting in net magnetization. These materials have a high magnetic susceptibility that is dependent on the magnetic field. At temperatures above the Curie temperature, they also exhibit Curie-Weiss behavior. The majority of ferrimagnetic materials are ceramics, which are excellent electrical insulators. Therefore, electrical losses in these materials are a lot lower than in metallic ferromagnetic materials. Figure

2.5 shows the magnetic moment of ferrimagnetism when there is an external magnetic field's absence and presence.

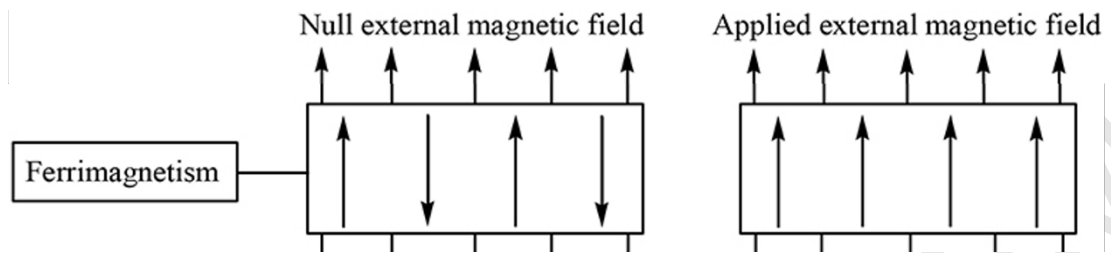


Figure 2.5: Magnetic moment of ferrimagnetism when there are absence and presence of external magnetic field (*Dia-magnetism*, n.d.)

2.3 Hysteresis Loop

Magnetic hysteresis is a property of ferromagnetic substances in which magnetic induction lags behind the decreasing magnetizing field. A hysteresis loop depicts a ferromagnetic material's magnetizing force, H against magnetic flux density, B . The hysteresis curve is specific to the studied material and might vary in size and form. Ferromagnetic materials receive energy in the form of work done to align the randomly oriented molecular dipoles along a particular direction when magnetized. However, when the magnetizing field is withdrawn, the molecular dipoles do not return to their initial locations entirely, and the specimen retains some magnetism. Hysteresis loss happens when there is definite energy loss in each cycle because the energy spent during magnetization is not entirely recovered after demagnetization. Examples of magnetic characteristics of a material that may be determined using the hysteresis loop are retentivity, residual magnetism, coercivity, permeability, and reluctance.

Figure 2.6 shows a hysteresis loop. Hysteresis loops begin at the origin (B and $H=0$). Before being magnetized, the specimen still portrays paramagnetism due to disoriented magnetic dipole. When a magnetizing force (H) is applied to an initially unmagnetized specimen, the magnetization curve (curve OAS) is increased until the saturation point. The direction of magnetic dipole moments is the same as the direction of the magnetic field at the saturation point (S). At point S, the magnetic flux becomes constant. Some residual magnetism remains when H progressively decreases to zero; the declining curve does not match the path of the initial curve. At $H=0$, B has a positive value, where this point is called the retentivity point (R). To eliminate the residual magnetization, a reverse field is used to decrease the magnetic induction to zero (path RB). The magnetic flux is equal to zero when the coercive force is applied due to the cancellation of dipole moments operating in opposing directions. This point is called the coercivity point (C). When the reverse magnetizing force is increased, the saturation occurs in the opposite direction until it reaches the saturation value (point S'). If the reverse field is removed and a positive field is applied, the loop gradually moves upward, passing the same negative magnitude of retentivity and coercivity value until it reaches its original saturation value (path S'CS).

Figure 2.6: Hysteresis loop (*Dia-magnetism*, n.d.)

The shape and size of the hysteresis loop indicate the magnetic properties of materials. Soft materials have greater susceptibility, permeability, and remanence (retentivity) values but smaller coercivity values than substances called hard materials. The hysteresis loss is less in soft materials than in hard materials. Thus, the hysteresis loop of different materials helps choose a suitable material for different magnetic appliances.

2.4 Structure of Spinel Ferrite

The chemical formula for spinel ferrites is MFe_2O_4 , where M stands for divalent metal ions such as Cu, Ni, Mg, and others. Spinel ferrites are one of the most important types of magnetic materials, with a wide range of applications. Spinel ferrites are magnetically soft and may be substituted for metallic magnets like Fe and layered Fe–Si alloys, but they perform better owing to their superior magnetic

properties (Snelling, 2005). They may also provide good electrical resistance and have low magnetic losses. Spinel ferrite has intriguing characteristics owing to its high electrical resistivity, high magnetic permeability, and the ability to adjust its properties across a wide range.

Spinel ferrite has a cubic structure and is made up of a densely packed arrangement of oxygen anions arranged in a face-centered cubic (FCC) lattice structure. Tetrahedrally coordinated sites are surrounded by four of the closest oxygen atoms, and octahedrally coordinated sites are surrounded by six of the nearest oxygen atoms. Normal spinel ferrites, inverse spinel ferrites, and random spinel ferrites are the general types of spinel ferrites. Figure 2.7 shows the spinel ferrite structure.

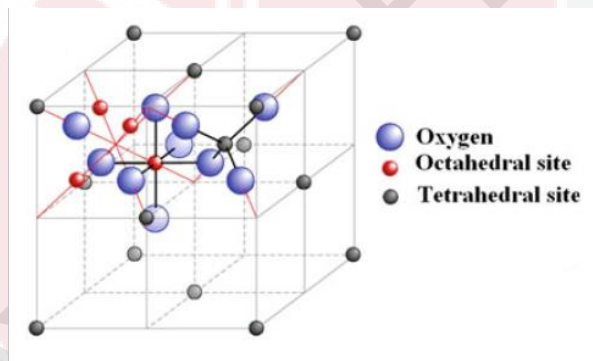


Figure 2.7: Spinel ferrite structure (Jadhav et al., 2020)

2.5 Cobalt Ferrite

Cobalt ferrite (CoFe_2O_4) is a hard magnetic material with a high coercivity of 5400 Oe, a moderate saturation magnetization of roughly 80 emu/g, excellent electromagnetic properties, and photomagnetism. It also has a high cubic magnetocrystalline anisotropy, is very magnetostrictive, has a high Curie temperature

T_C (520 °C), is chemically stable, and has outstanding mechanical hardness, wear resistance, and electrical insulation.

CoFe_2O_4 has a face-centered cubic crystalline structure with a large unit cell having eight formula units. Cobalt ferrite (CoFe_2O_4) is an inverted spinel ferrite with the majority of Co^{+2} ions in octahedral sites and Fe^{+3} ions in tetrahedral and octahedral sites. Divalent cations that occupy the tetrahedral site and trivalent cations with a significant attraction to the octahedral site are the principal regulators of these AB_2O_4 groups of ferrites. Hopping conductivity and magnetic coupling are formed when two transition metal ions are present at the same crystallographic position (Houshiar et al., 2014).

Fe^{3+} ions occupy tetrahedral sites, whereas Co^{2+} and Fe^{3+} ions share octahedral sites equally. The inverse spinel structure is a form of spinel structure. The CoFe_2O_4 material is often described as $(\text{Co}_x\text{Fe}_{1-x})[\text{Co}_{1-x}\text{Fe}_{1+x}] \text{O}_4$, where x is determined by thermal treatment and preparation conditions. Because the super-exchange interaction between tetrahedral Fe^{3+} ions and octahedral Fe^{3+} ions is usually different than the interaction between the tetrahedral Co^{2+} ion and Fe^{3+} ion, the difference in magnetic properties of the oxides, even though the compound has the same chemical composition, is due to variation of the cation distribution over tetrahedral and octahedral.

2.6 Co-precipitation Method

The co-precipitation technique is a frequently used ferrite synthesis process that produces a mixed precipitate comprising two or more insoluble components that

are precipitated simultaneously. Precipitates obtained through precipitation have wide size distribution, uncontrolled morphology, and agglomeration; due to the lack of control of chemical precipitation's reaction kinetics and nucleation and growth phase process. The solubility of various metal hydroxides varies with pH, and the cation ratios in the precipitate might differ from those in the solution (Valenzuela, 2005). Metal ion solutions are added dropwise and slowly to alkaline solutions to solve this problem (McColm & Clark, 1988). The pH of suspension must be raised higher than 10 to avoid the difference in the composition of precipitation if an alkaline solution is used (Takada, 1982). Moreover, correct control of experimental factors such as pH, reactant and ion concentrations, and temperature is crucial as they impact the precipitation process (Ashik et al., 2015).

One of the benefits of using the co-precipitation method of producing homogeneous nanomaterials with tiny size and size distribution directly from the solution via various chemical reactions. The co-precipitation method is most likely the most straightforward chemical approach for producing magnetic nanoparticles. The co-precipitation technique has several advantages: quick and straightforward preparation, easy control over particle size and composition, and energy efficiency (Bhagyaraj, 2018). The co-precipitation method is developed due to its low cost, simplicity, nontoxic precursors, industrial-scale yields, and short time (Saputro, 2019).

2.7 Composite

Composites are materials manufactured by mixing two or more elements with distinct physical or chemical characteristics that are stronger to provide a superior end product. Composites are generally created with a specific use in mind, such as

increased strength, efficiency, or durability, because of the material's unique nature and our capacity to modify the product's features by adjusting their components. As a result, composites have numerous favorable physical characteristics and provide a high degree of design freedom.

One of the key reasons for using composite materials instead of traditional components is to save weight. Some composites are lighter than their starting materials, but they may also be more durable. Another advantage of using composites over traditional materials is their heat and chemical resistance, as well as their electrical insulating properties. Composites may have a variety of properties that are seldom seen in a single substance compared to traditional materials.

2.8 Optical Band

The excitation of electrons from the valence band to the conduction band with photons of a certain frequency is used to determine the optical band gap. The number of carriers involved in the process is unaltered, and the total number of carriers in the semiconductor stays constant. Electrons in atoms and molecules commonly have different energy levels. However, in solids, these energy levels combine to form bands. The valence energy band contains the electrons that bind the atoms together, whilst the remaining electrons that are not required for bonding are able to move freely around. These may be present in the conduction band and may take part in electrical conduction (Kittel & McEuen, 2019).

The conduction and valence bands can either overlap or not, depending on the material. The gap between conduction and valence bands called the energy band gap is the minimum excitation energy for electrons to cross from the valence band to the

conduction band. If the electron is given enough energy for bonding, the electron can be excited to cross the conduction gap. The properties of the material depend on the size of the band gap. The electrical properties are determined from the presence of electrons in the conduction band.

There is overlap between conductors' conduction and valence bands; therefore, the electron can populate the conduction band. There is no overlapping between the conduction and valence bands in insulators, and there is a large energy gap between both bands. Therefore, a large amount of energy is needed to excite the electron from the valence band to the conduction band. So, the electron cannot cross to the conduction band. In semiconductors, there is a gap, but it is small enough for an electron to excite from the valence band to the conduction band if enough energy is applied to the semiconductor. Figure 2.8 shows the band diagrams for a conductor, semiconductor, and insulator.

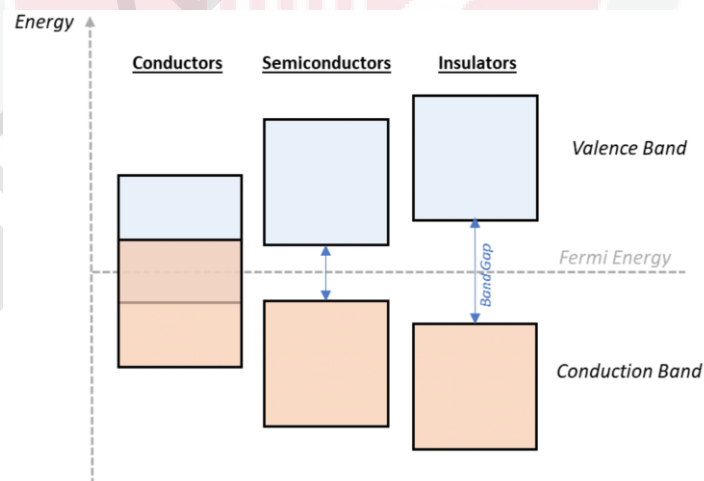


Figure 2.8: Band diagrams for a conductor, semiconductor, and insulator (Maxmatch, 2019)

2.9 Electrical Properties

The electrical properties of a substance determine its capability to conduct electrical current. Some of the electrical properties include electrical resistivity and electrical conductivity. Electrical conductivity determines how well a material allows the flow of an electric charge. Conductivity It is the ratio of current density over electric field strength. The symbol for electrical conductivity is σ (sigma). The SI unit of electrical conductivity is siemens per meter (S/m). High conductivity denotes a substance that easily facilitates the passage of electric current. Electrical resistivity is the reciprocal of electrical conductivity. Resistivity is a material property that opposes the passage of electric current through a substance. Electrical resistance is given in Ohms.

CHAPTER 3

METHODOLOGY

3.1 Introduction

All experimental data used in this research came from the previous final year project student. Therefore, this chapter provides the procedure taken to prepare cobalt ferrite nanocomposites using co-precipitation method, including the chemical reaction of raw materials, the specific synthesis methods, and obtaining characterizations. The co-precipitation methods involve weighing raw material, mixing, filtering, drying, grinding, and sintering. The sample produced was mixed with carbon black with different weight percentages of carbon black of 2%, 4%, 6%, 8%, and 10% to produce nanocomposites. A detailed explanation of the characterization process done on the nanocomposites to measure and determine the properties of the nanocomposites, which are the structural, morphological, optical, magnetic, and electrical properties, is also discussed in this chapter. The properties and characterization of the nanocomposites were analyzed by using XRD, FESEM, Two Point Probe, VSM, and UV-Vis. Figure 3.1 shows the flowchart of sample preparation.

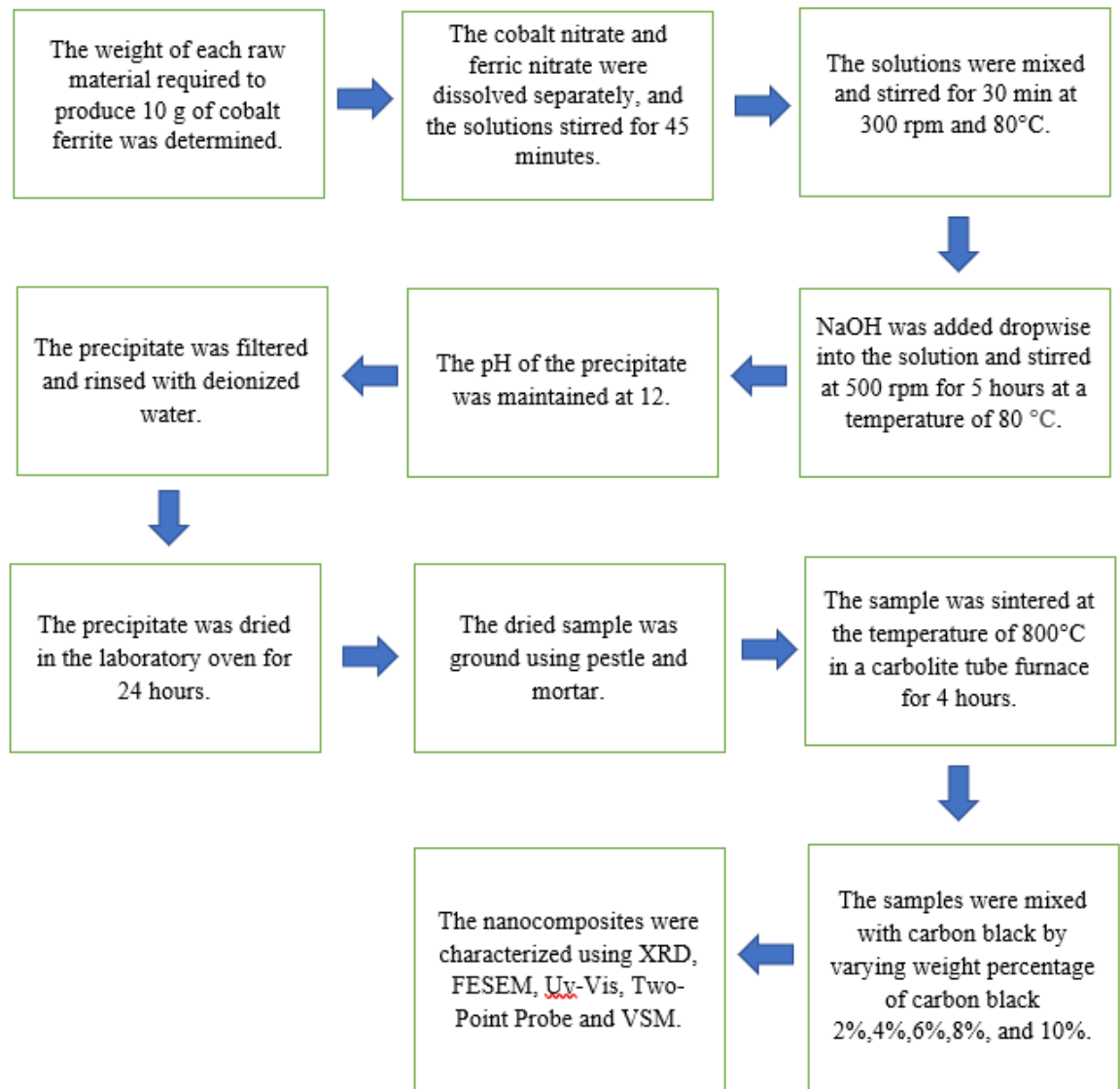


Figure 3.1: Flowchart of sample preparation (Juliza Samsor, 2020)

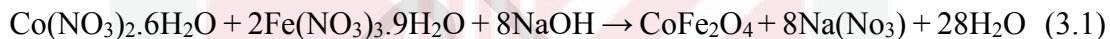
3.2 Experimental Procedure

3.2.1 Raw Materials

The co-precipitation method was used to synthesize cobalt ferrite nanoparticles. The cobalt nitrate salt, $\text{Co}(\text{NO}_3)_2$, and ferric nitrate salt, $\text{Fe}(\text{NO}_3)_3$, were used as the metal precursors in the co-precipitation process. The precipitating agent used is sodium hydroxide, NaOH . Deionizing water was used to dissolve the salt and to filter and rinse the sample precipitate.

3.2.2 Weighing

The weight of each raw material required was determined from the balanced chemical equation stated in Equation 3.1:



The weight of raw materials needed to produce 10 g of cobalt ferrite nanoparticles was obtained by using stoichiometry and balanced equations. The calculation of the weight of raw materials is shown in Appendix A. The raw material was weighted using the electronic analytical balance shown in Figure 3.2. Some precautions were taken to decrease the error in measurement. The filter paper was placed on the analytical balance, and the weight of the paper was cancelled out to display reads 0.0000 g. The weighted material was placed on the center of the weighing pan. The weighing area of the electronic analytical balance was cleaned by using a soft brush to remove spilled materials before and after every measurement.

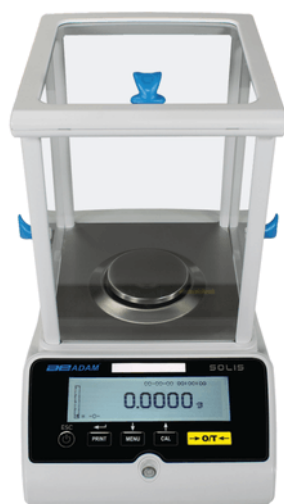


Figure 3.2: Electronic analytical balance (Adam Equipment, 2018)

3.2.3 Mixing

The cobalt nitrate and ferric nitrate nonahydrate were dissolved separately with deionized water. Then the solutions were put under stirring using a magnetic stirrer and hotplate. The solutions were stirred for 45 minutes for each solution. Next, all the solutions were mixed to form a homogeneous solution, and the mixed solution was stirred for 30 min at 300 rpm and 80°C. The precipitating agent used in this process is an aqueous solution of sodium hydroxide, NaOH. NaOH solution was added dropwise from a burette to mixed solution while stirred continuously with a magnetic stirrer and hotplate. During the titration process, the solution was mixed at 500 rpm for 5 hours at a constant temperature of 80 °C. The addition of NaOH solution was regulated to maintain a constant pH of 12 to control the nucleation (Allaadini & et al., 2014). The particle size of the sample synthesized by the co-precipitation method strongly depends on the pH of the precipitation medium and the molarity of the precursor. The particle size decreases as the pH values increase as there is an increase in molarity of the solution (P. & et al., 2016). The precipitate was cooled to room temperature after

the stirring was finished. Figure 3.3 shows the hotplate and magnetic stirrer used in the mixing process.



Figure 3.3: Hotplate and magnetic stirrer (Home Science Tools, n.d.)

3.2.4 Filtering and Rinsing Process

The formed precipitate was filtered by using filter paper and a filter funnel. The precipitate was rinsed with deionized water to remove the by-product solution from the precipitate. A small amount of water was used to dissolve the deionized one at a time to avoid losing the precipitate. After rinsing, the precipitate was dissolved in deionized water and filtered again. The process was repeated a few times until the filtered solution reached pH=7. Figure 3.4 shows the process of filtration.

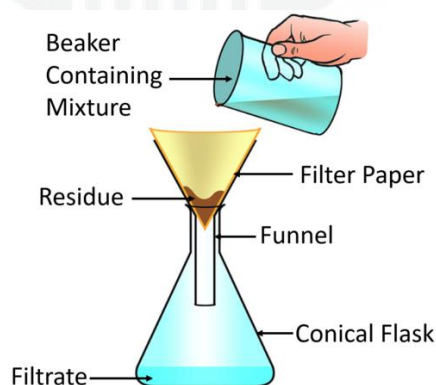


Figure 3.4: Filtration process (Key Stage Wiki, n.d.)

3.2.5 Drying

The solid precipitate was put into the laboratory drying oven for 24 hours at a temperature of 95°C. The drying process is essential to remove residual traces of rinse solution and remove any volatile impurities.

3.2.6 Grinding

The dried sample was ground using pestle and mortar, as shown in Figure 3.5. Tools were wiped clean of dust surface before the dried sample was grinding to minimize the effect on the analysis results. The time to grind the sample was about 45 minutes until a fine powder was obtained.



Figure 3.5: Pestle and mortar (MxRady, n.d.)

3.2.7 Sintering

Sintering is a thermal treatment that adds strength and integrity to the applied powder compact. The sintering temperature used should be lower than the melting point of the main component of the Powder Metallurgy material. After grinding, the

sample of cobalt ferrite was sintered at the temperature of 800°C in a carbolite tube furnace, as shown in Figure 3.6 for 4 hours. The sample was placed in an alumina boat, and then the alumina boat was put inside the carbolite tube furnace. The time taken to increase the temperature to 800°C was 5 hours. Once the temperature reached 800°C , the constant temperature was maintained for 4 hours. Then the time taken to decrease the temperature was 5 hours. Figure 3.7 shows the graph of sintering time to prepare cobalt ferrite nanoparticles.



Figure 3.6: Carbolite tube furnace (Keison, n.d.)

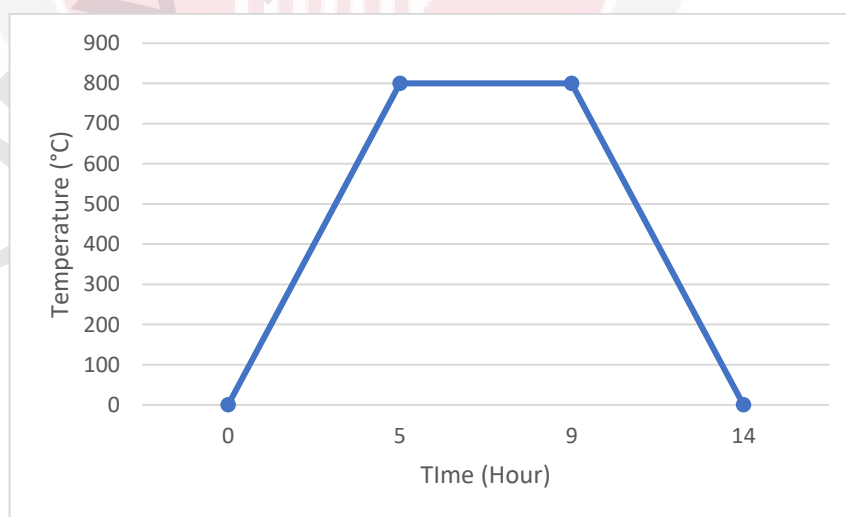


Figure 3.7: Graph of sintering time to prepare Cobalt Ferrite nanoparticles

3.2.8 Composite Preparation

The cobalt ferrite nanoparticles were mixed with carbon black by varying the weight percentage of carbon black, which were 2%, 4%, 6%, 8%, and 10%. The sample is denoted as CoF for pure cobalt ferrite, CB for carbon black, and CoFCB2%, CoFCB4%, CoFCB6%, CoFCB8% and CoFCB10% for cobalt ferrite/carbon black with varying weight percentage. The cobalt ferrite nanoparticle and carbon black were mixed using mortar and pestle. Each sample was mixed thoroughly for about 1 hour to ensure the composite was mixed well. The weight percentage and mass of components in each composite sample are shown in Table 3.1.

Sample	Weight Percentage (%)		Mass (g)	
	Cobalt Ferrite	Carbon Black	Cobalt Ferrite	Carbon Black
CoF	100	0	4.00	0.00
CoFCB2%	98	2	3.92	0.08
CoFCB4%	96	4	3.84	0.16
CoFCB6%	94	6	3.76	0.24
CoFCB8%	92	8	3.68	0.32
CoFCB10%	90	10	3.60	0.40
CB	0	100	0.00	4.00

Table 3.1: Weight percentage and mass of cobalt ferrite and carbon black in each sample

3.3 Characterization

The characterizations of cobalt ferrite/carbon black nanocomposite were done by the previous final year project student are structural, morphology, electrical properties, optical properties, and magnetic properties. X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), Ultraviolet-visible (UV-Vis) Spectroscopy, Two-Point Probe, and Vibrating-Sample Magnetometer (VSM) were used to characterize the nanocomposite.

3.3.1 X-Ray Diffraction (XRD)

X-ray diffraction which is PW 3040/60 MPD X'Pert High 25 Pro PAN-Analytical from Philip shown in Figure 3.8, is used to determine materials' structural characterization. Compared to other analytical techniques, powder diffraction provides fast results and non-destructive analysis of multi-components without extensive sample preparation (Han, 2008). X-ray diffraction produces different peak locations and intensities corresponding to a specific crystal phase. The width and intensity of a diffraction peak are determined by the size and shape of the unit cell and are independent of atomic positions inside the cell. Each peak represents a lattice plane and is characterized by the Miller Index.



Figure 3.8: X-Ray Diffraction Instrument

When an incident beam of monochromatic X-ray is directed toward the sample, the atoms will cause the incoming X-rays to scatter into many specific directions, and diffraction occurs. The scattered x-rays interfere constructively at a certain angle and produce intense reflected x-rays. When the conditions fulfill Bragg's Law, constructive interference of incidents occurs. Bragg's law established the general relationship between the wavelength of the incident beam, incidence angle, and interplanar distance of crystal. Bragg's law, denoted by Equation (3.2), is the fundamental law of diffraction.

$$n\lambda = 2d \sin \theta \quad (3.2)$$

Where n is the order of reflection (integer), λ is the wavelength of the incident beam, d is the interplanar distance of crystal, and θ is the incidence angle.

3.3.2 Field Emission Scanning Electron Microscopy (FESEM)

Field Emission Scanning Electron Microscopy (FESEM) used was FEI NOVA NanoSem 230 shown in Figure 3.9 is a microscope that uses electrons to see small topographic features on the surface of materials as small as 1 nm. FESEM can give qualitative information of the specimen, including its topography, morphology, composition, and structural information.



Figure 3.9: Field Emission Scanning Electron Microscopy (FESEM)

Electrons are driven through a significant electrical field gradient after being released from a field emission source. In a zig-zag pattern, electrons scan the substance. The initial electrons are focused and deflected by electronic lenses inside the high vacuum column, resulting in a focused beam that bombards the target. As a consequence, secondary electrons are ejected from the spots of each object. The surface structure of the item influences the angle and velocity of these secondary electrons. A detector captures the secondary electrons and creates an electrical signal. This signal is amplified and turned into a video scan image that may be seen on a monitor or a digital image that can be saved and processed further.

3.3.3 Vibrating-Sample Magnetometer (VSM)

A vibrating sample magnetometer (VSM) as shown in Figure 3.10 is a scientific device used to test magnetic characteristics such as hysteresis loop, permeability, and others. Based on Faraday's Law of Induction, the vibrating component produces a changing magnetic field of the sample, which creates an electrical field in a coil. According to Faraday's rules of magnetic induction, an alternating current voltage generated in the electrical device is proportional to the rate of change of magnetic flux in the circuit. Hence, the applied magnetic field also influences the magnitude of the moment within the sample. As the sample vibrates vertically near the detecting coil, an alternating current signal is created at a frequency determined by the sample oscillation.



Figure 3.10: Vibrating sample magnetometer (VSM)

3.4 Ultraviolet-visible (UV-Vis) Spectroscopy

UV-Vis spectrophotometry is a method for measuring light absorption in the ultraviolet and visible parts of the electromagnetic spectrum with a wavelength range of from 190 nm to 1,100 nm. The concentration of a specific chemical component may be estimated by measuring the number of photons (light intensity) that reach the detector by using a spectrophotometer and absorption/transmission measurements. The UV-Vis Spectroscopy used was Shimadzu UV-3600. The UV-Visible Spectroscopy Principle is based on the absorption of ultraviolet or visible light by chemical molecules, resulting in distinct spectra. The process of excitation and de-excitation of the atom due to the absorption and release of a distinct amount of energy causes the UV-Vis spectrum. Incident light may be absorbed, reflected, or transmitted when it impacts materials. The absorption of UV-Vis light induces atomic excitation, which is the transition of atoms from a ground state to an excited state. A certain amount of energy needs to be absorbed by the electron to enable it to transit to a higher orbital level. The shorter band gaps mean that the light is absorbed at shorter wavelengths. The energy needed for molecules to execute these transitions is electrochemically different. Figure 3.11 shows the schematic design of the UV-Vis setup.

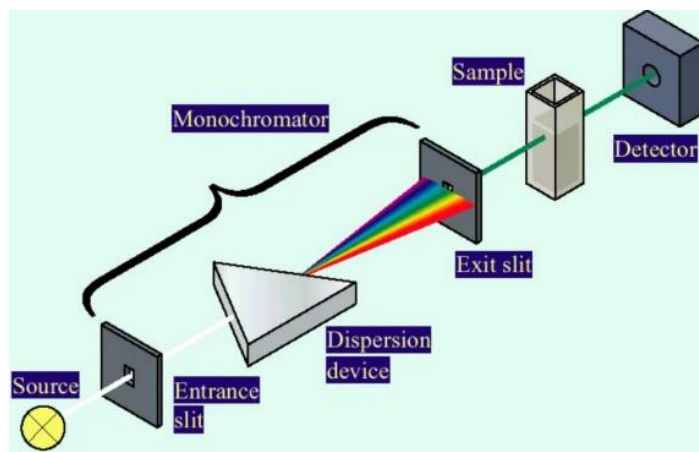


Figure 3.11: Schematic diagram of UV-Vis setup

3.3.5 Two-Point Probe

A Two-Point Probe is used to obtain the material's resistance by measuring the current and potential difference across the sample pellet. Figure 3.12 shows the schematic diagram of the two-point probe measurement. Before measuring the samples' resistivity, samples were pelletized using a manual hydraulic press. The mass of each pellet of the sample is 0.4 g. The thickness and diameter of each pellet are measured using a micrometer screw gauge. Each probe was placed at the sides of the pellet where one of the probes supplies current while the other provided a voltage probe measured by a multimeter. The two-point probe is used due to the simple usage, but measurement error could occur if used on a low resistance material as the wire resistance can affect the overall resistance (Wisung, 2018).

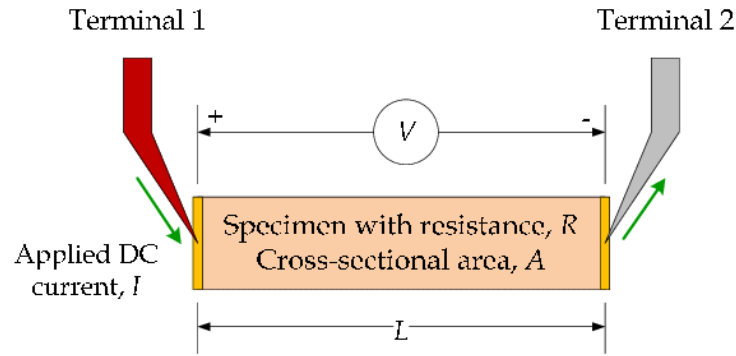


Figure 3.12: Schematic diagram of Two-point probe measurement

An Ohm's Law graph, a graph of current versus voltage, I - V , is plotted to calculate the value of resistance from the slope of the graph. Ohm's Law (Equation 3.3) was used to calculate the resistance value.

$$V = IR \quad (3.3)$$

V is the potential difference across the material, I is the current, and R is the resistance.

The resistivity of the sample was calculated by using equation 3.4:

$$\rho = \frac{RL}{A} \quad (3.4)$$

Where ρ is the resistivity of the material, R is the resistance, L is the thickness of the material, and A is the cross-sectional area of the material.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

All experimental data discussed in this chapter came from the previous final year project student. This chapter provides the results and discussion of the characterization of the structural, morphological, optical band gap, electrical and magnetic properties obtained using XRD, FESEM, UV-Vis Spectroscopy, Two-Point Probe and VSM.

4.1 XRD Analysis

The crystalline nature and phase formation of cobalt ferrite and carbon black composite with different weight percentages of carbon black which prepared by co-precipitation method were characterized using PW 3040/60 MPD X'pert High PRO Panalytical Diffractometer with Cu K α radiation ($\lambda=1.540598$ Å) in the 2θ range 20 - 80°.

Figure 4.1 shows the XRD pattern of CoF, CB, and CoFCB nanocomposites. From Figure 4.1, the XRD pattern of CoF has the peaks at $2\theta=30.34^\circ$, 35.70° , 37.34° , 43.34° , 53.72° , 57.26° , 62.84° , and 74.30° , which indicates the planes respectively at (022), (113), (222), (004), (224), (115), (044) and (335). These values match with the reference data (ICSD 160059) of CoFe₂O₄. The crystal structure of CoF is a spinel cubic structure with a space group of Fd-3m. Meanwhile, no peaks from other crystalline phases or impurities can be identified, suggesting the high purity of CoFe₂O₄. The XRD pattern of CB had the peaks at $2\theta= 26.72^\circ$ and 43.14° which correspond with (002) and (100) planes which match with the research from Ungár et

al. (2005). The peaks of the XRD pattern of CB are broad, indicating the amorphous carbon black content.

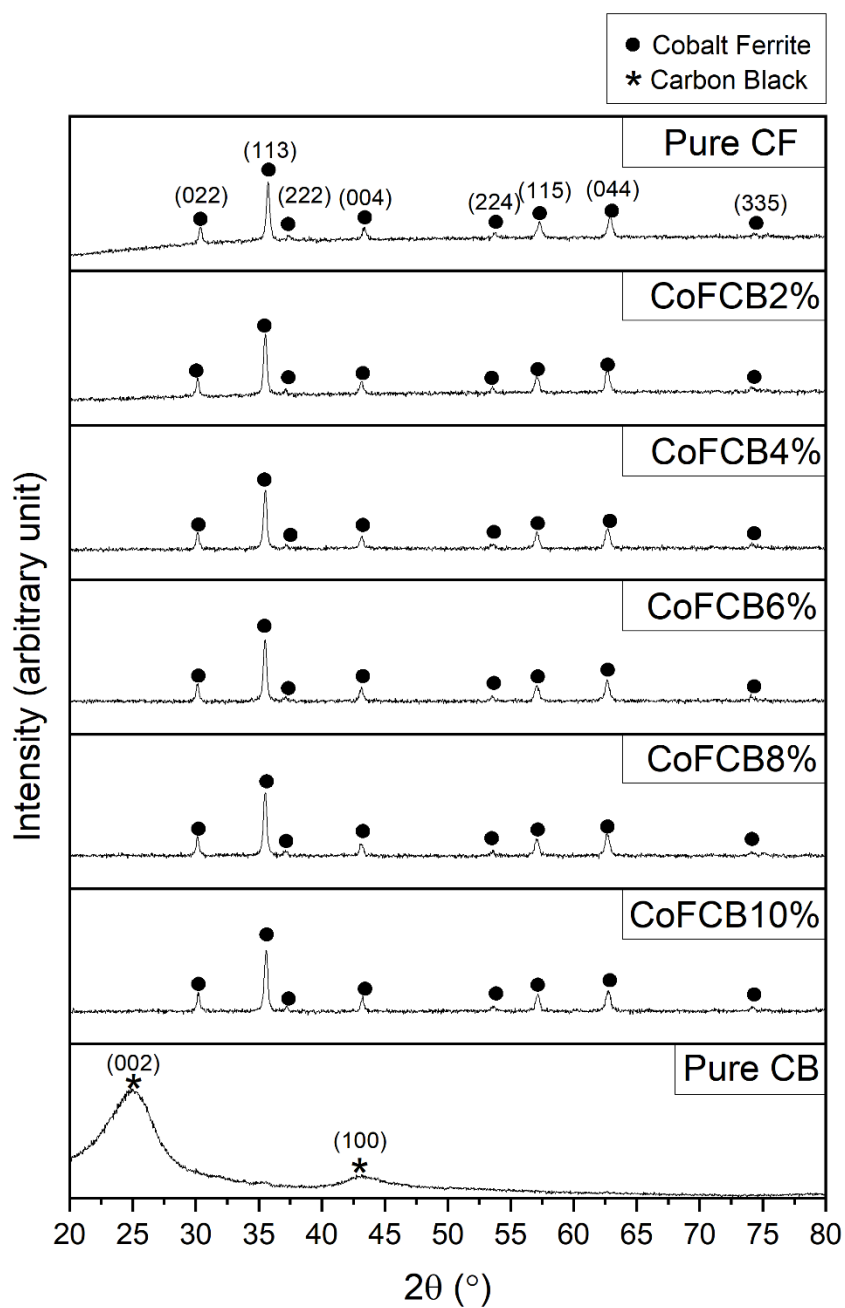


Figure 4.1: XRD Pattern of CoF, CB and CoFCB nanocomposites with varying weight

From the XRD patterns of the CoFCB nanocomposites, it was observed that the single spinel phase is formed in the nanocomposite. The peaks of the XRD pattern of CoFCB nanocomposite are narrower than the peaks of the XRD pattern of the pure CoF, which indicates an intensification in crystallinity. The secondary phase is not showing in the XRD pattern as there is no visible peak of CB in the XRD pattern of CoFCB nanocomposites, which is most likely due to the amorphous nature of the CB and the low weight percentage of CB in the nanocomposite. The baseline of the XRD patterns becomes more straightened as the weight percentage of CB increases. There is also some shifting of peaks to lower angles with the addition of CB. The position shift of the XRD peaks of the CoFCB nanocomposite compared to pure CoF might be due to the changes in lattice constant or the structure of the sample (Desai, 2002).

The crystallite size of cobalt ferrite and carbon black are calculated using the Scherrer equation (Equation 4.1):

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (4.1)$$

Where D is crystallite size in nm, K is Scherrer's constant, which is 0.9, λ is the wavelength of the X-ray sources, which is 0.15406 nm, β is the full width at half maximum main diffraction peak in radian, and θ is the Bragg angle. The average crystallite size of cobalt ferrite and carbon black is determined by considering the highest peak of the XRD pattern (Saputro, 2019), which is from the (113) and (002) planes were 30.91 nm and 3.48 nm, respectively.

4.2 Morphological Properties

The surface morphology and particle size distribution of Cobalt Ferrite, Carbon Black, and Cobalt Ferrite/Carbon Black nanocomposite were observed using Field Emission Scanning Electron Microscopy (FESEM). FESEM was used to visualize the microstructure and topographic image on the surface. The average particle size of the samples was calculated from FESEM micrographs by considering at least 100 particles.

Figure 4.2 and Figure 4.3 show that FESEM image and histogram particle size distribution for pure CF and pure CB. From Figure 4.2, CoF nanoparticles are cubically spherical shaped, and it can be observed from the surface of the sample that the size of ferrite particles is uniform and has fine particles. There are some agglomerations on the surface of CoF might be due to the interaction of magnetic nanoparticles. From Figure 4.3, the particle size distribution of CoF nanoparticles ranges from 50-110 nm with an average particle size of 73 nm. At the same time, the particle size of carbon black ranges from 40 nm to 100 nm, with an average particle size of 64 nm.

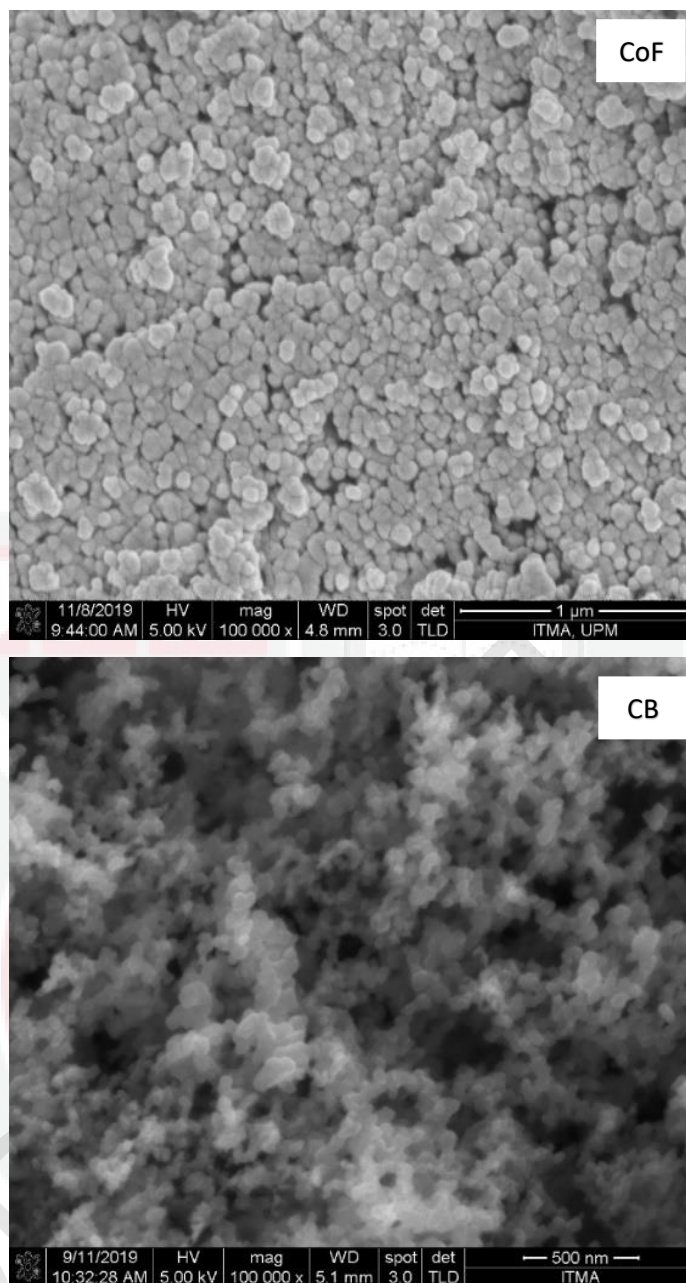


Figure 4.2: FESEM Micrographs of CoF and CB

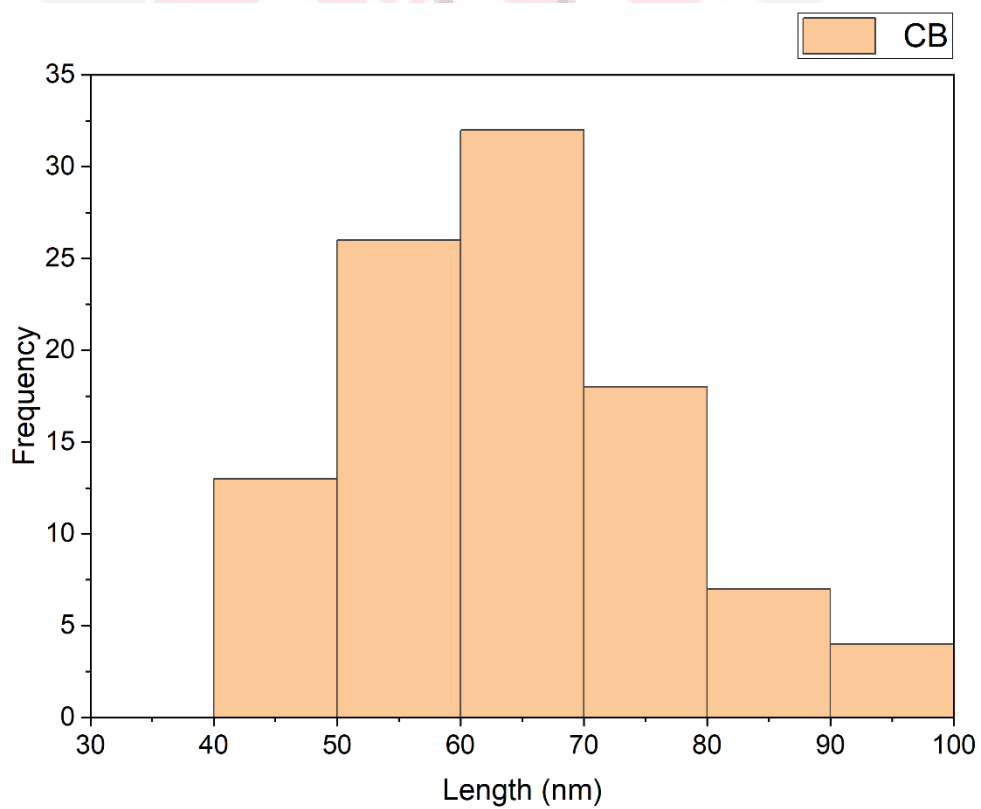
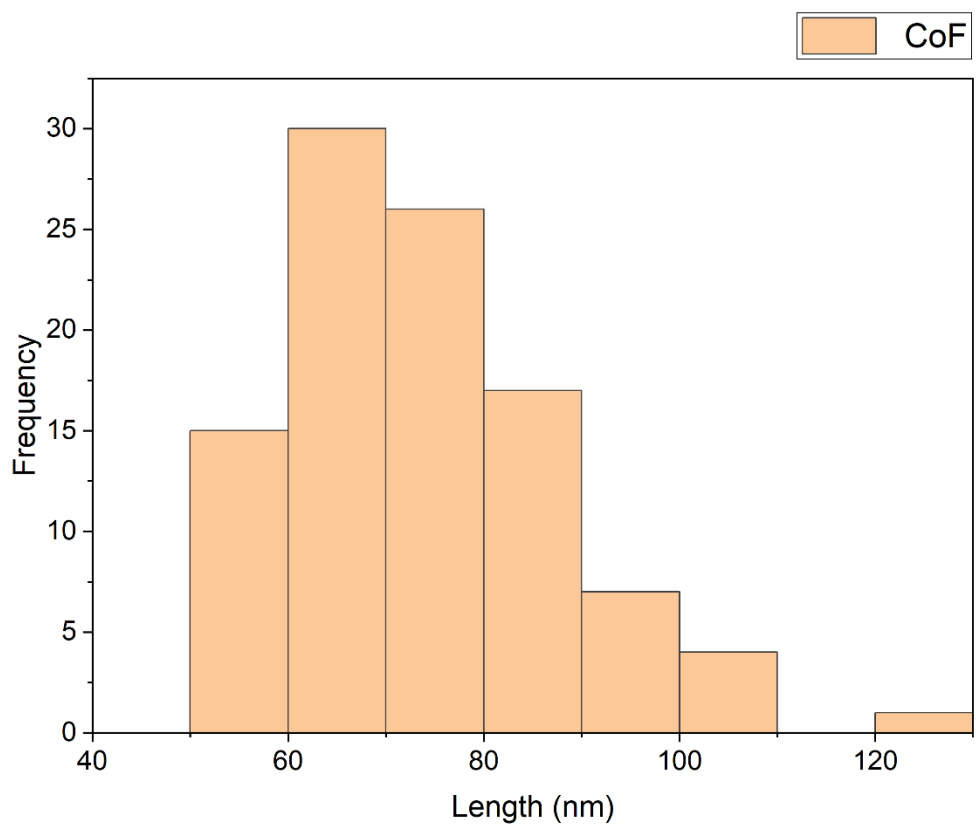


Figure 4.3: Particle Size Distribution Histogram of CoF and CB

From the FESEM micrograph of CoFCB nanocomposites in Figure 4.4, it is hard to distinguish between cobalt ferrite and carbon black nanoparticles because it only revealed the surface morphology of the samples. Therefore, Electron Dispersion X-Ray (EDX) was used to confirm the presence of the incorporation of carbon black nanoparticles in CoFCB nanocomposites. Energy Dispersive X-Ray Analysis (EDX) is an x-ray technique used to determine the elemental composition of materials. The elemental analysis data consist of spectra with peaks corresponding to the elements that make up the actual composition of the analyzed material. The elemental analysis of CoF, CB, and CoFCB nanocomposite with different weight percentages of carbon black nanocomposite was recorded for each sample using EDX, as shown in Figure 4.5. From the EDX analysis of pure CoF, CoF contains the elements of Co, O, and Fe. In CB, it shows that the only element present in the sample is C. For the EDX analysis of CoFCB nanocomposites, all samples show the presence of elements Co, O, Fe, and C, which validates the presence of the carbon black in the CoFCB nanocomposites. As the carbon black content in the CoFCB nanocomposite increases, the peak of C in the EDX spectrum increases, which indicates the increasing carbon black content. The EDX sample shows no foreign element, which means there is no impurity inside the sample of CoF, CB and CoFCB with varying carbon black content.

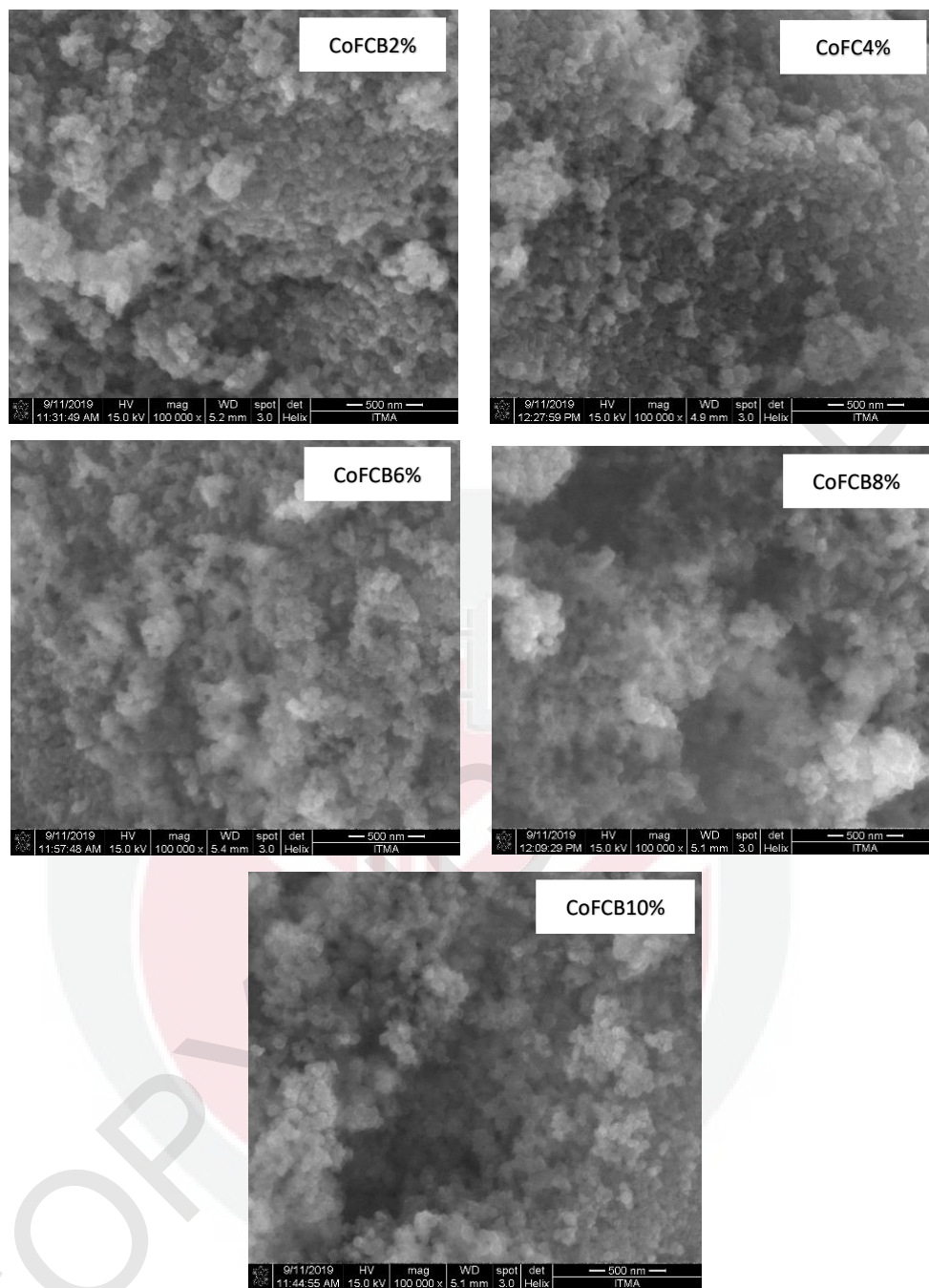


Figure 4.3: FESEM micrograph of CoFCB nanocomposites at 100k magnification

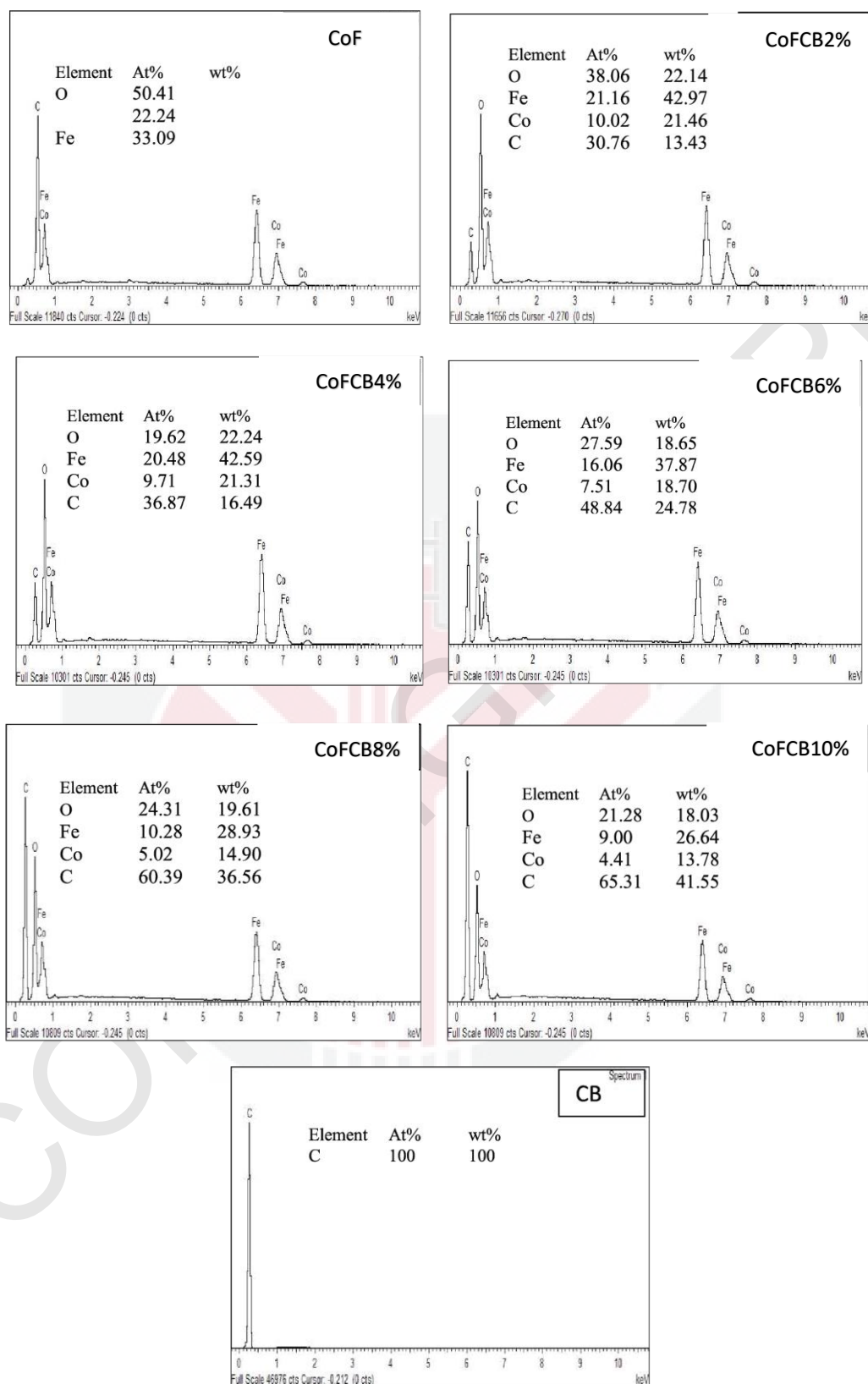


Figure 4.5: EDX Spectra of CoF, CB and CoFCB nanocomposites

4.3 Optical Band Gap

Absorbance and reflectance measurements of CoF, CB, and CoFCB nanocomposites were obtained in the wavelength ranges from 220 to 800 nm by using ultraviolet-visible spectroscopy (UV-Vis). Figure 4.6 is the graph of absorbance against wavelength of the nanocomposite, while Figure 4.7 is the graph of reflectance against wavelength of the nanocomposite.

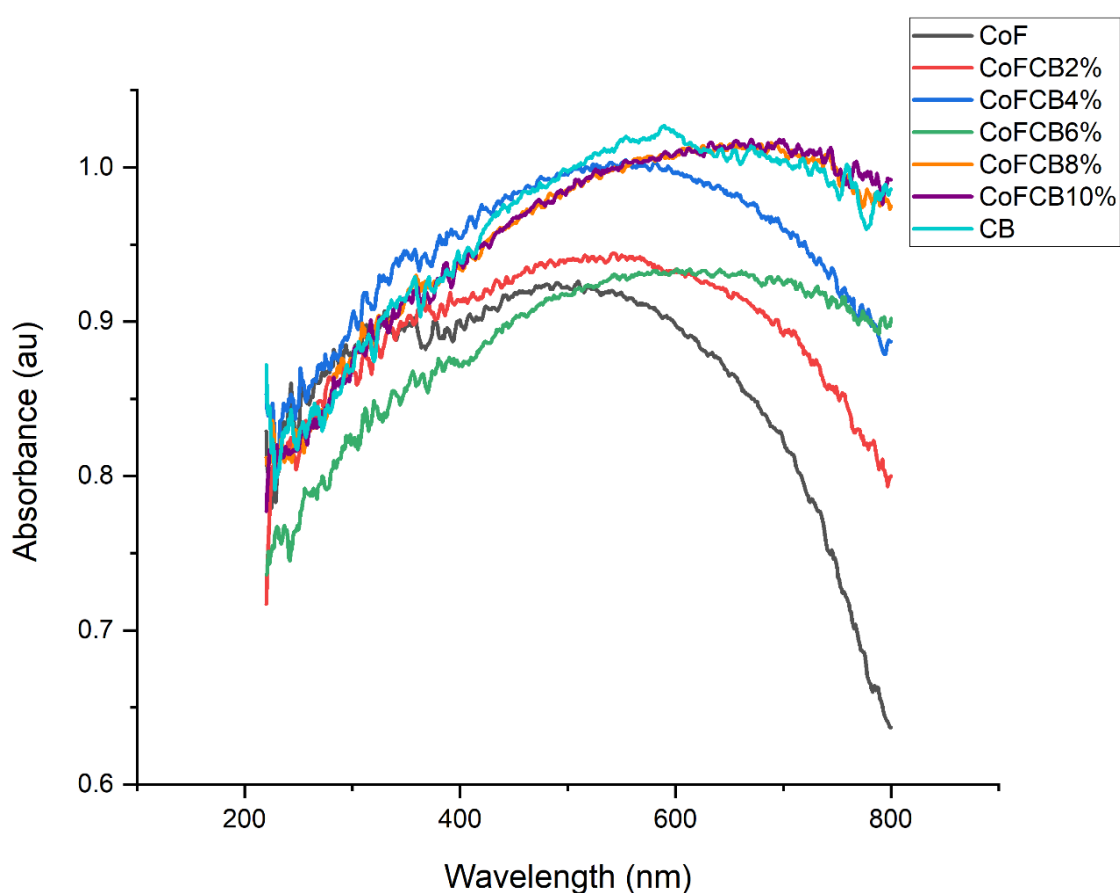


Figure 4.6: Graph of absorbance versus wavelength of CoF, CB and CoFCB nanocomposites

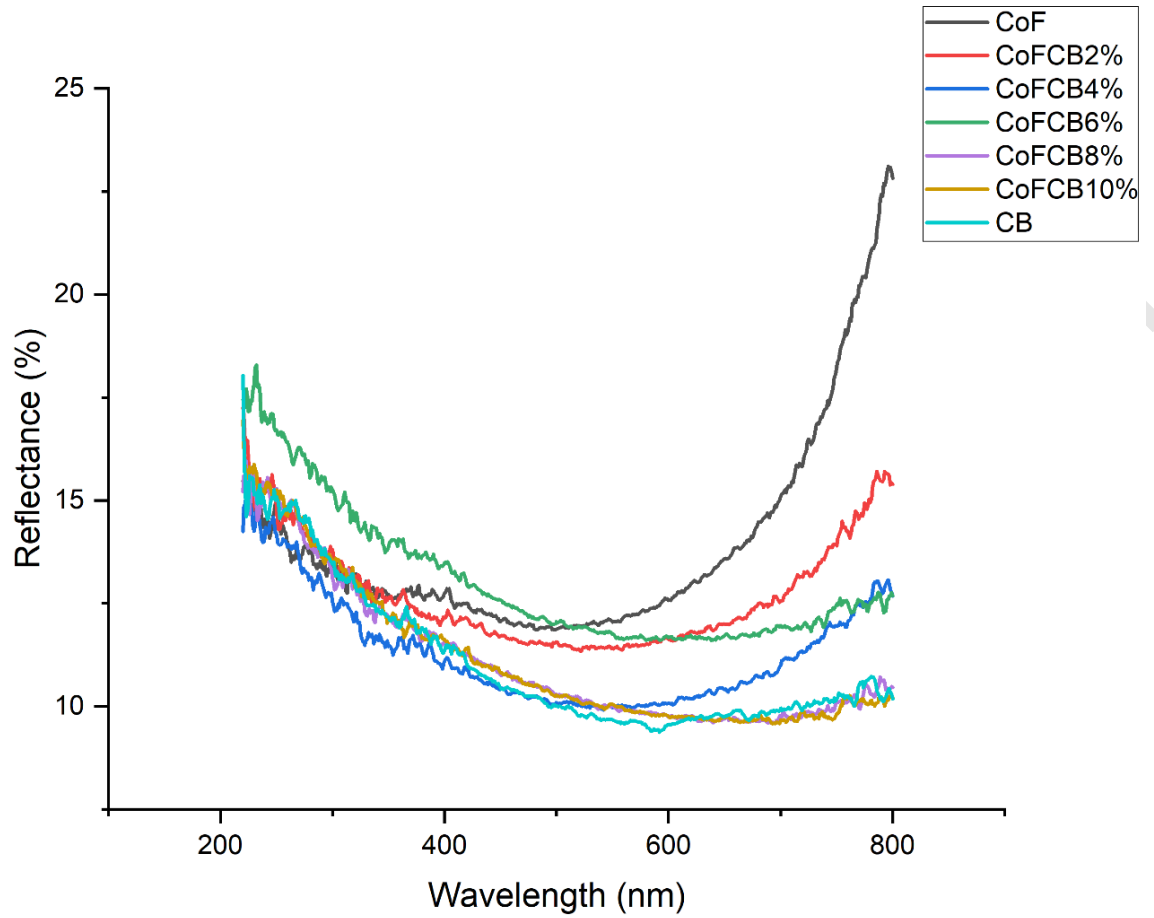


Figure 4.7: Graph of reflectance versus wavelength of CoF, CB and CoFCB nanocomposites

The absorbance measurements gained were used to study cobalt's effect on the optical band gap energy. The optical band gap energy can be estimated from UV-Vis absorbance data by extrapolating the line of the Tauc plot graph. The Tauc plot method is based on the Tauc equation, which is Equation 4.2, is used to calculate the absorption edges from absorption data.

$$(\alpha h\nu)^n = K(hf - E_g) \quad (4.2)$$

Where α is the absorption coefficient, h is Planck's constant, f is the photon's frequency or $E=hf$ which is the incident photon energy, K is an energy independent

constant, E_g is the band gap energy. The exponent "n" represents the nature of electron transition. For allowed direct band gap energy, $n=2$ while $n=1/2$ for indirect allowed band gap energy. α is calculated by using equation 4.3 derived from the Beer-Lambert Law.

$$\alpha = A \frac{1}{\log(e)L} \quad (4.3)$$

Where A is the absorbance, $\log(e) = 0.4343$, and L is the standard cuvette pathlength or the distance that light passes through the sample in the cuvette, $L = 1 \text{ cm}$.

The n-value is determined by plotting the graph of $(\alpha h\nu)^n$ against $(h\nu)$ with different n-values to see which one produces the greatest fit and so indicates the right transition type, with the energy-axis intercept indicating the band gap value (Coulter & Birnie, 2017). The n-value is determined to be 2 due to the graph $(\alpha h\nu)^2$ versus $(h\nu)$ which is plotted to have the best fit. After plotting the graph, the optical band gap energy is estimated from the energy-axis intercept made by the extrapolating liner part of the curve. The extrapolated graph of $(\alpha h\nu)^2$ versus $(h\nu)$ is stated in Appendix B.

Table 4.1 shows the estimated band gap energy of CoF, CB and CoFCB nanocomposites. CoF's optical band gap energy is 1.36 eV, which is lesser than the optical band gap energy of 1.92 eV from the research of Annie et al. (2016) but the highest among the other samples. The lowest optical band gap energy is CB's which is 1.01 eV. This result shows that CoF has the lowest conductivity while CB has the highest conductivity among the samples. The band gap energy decreases from 1.24 eV to 1.04 eV with increasing carbon black concentration in cobalt ferrite/carbon black nanocomposites shown in Table 4.1. The band gap is controlled by several variables, including crystallite size, impurity content, and structural characteristics (Kale &

Lokhande, 2004). All the samples can be concluded as semiconductors as the band gap energy is lesser than 3.0 eV.

Table 4.1: Optical band gap energy of CoF, CB and CoFCB nanocomposites

Sample	Band gap energy (eV)
CoF	1.36
CoFCB2%	1.24
CoFCB4%	1.16
CoFCB6%	1.09
CoFCB8%	1.06
CoFCB10%	1.04
CB	1.01

4.4 Electrical Properties

The readings of voltage, V and current, I across CoF, CB and CoFCB nanocomposites in pellet form were measured using a two-point probe at room temperature. The resistance of the sample is the reciprocal of the slope of the plotted graph of I-V shown in Appendix C. Conductivity, σ is gained from the resistivity as the conductivity is the reciprocal of the resistivity which is Equation 4.4.

$$\sigma = \frac{1}{\rho} \quad (4.4)$$

Table 4.2 show the resistivity and conductivity values of CoF, CB and CoFCB nanocomposite. The resistivity of CoF nanocomposite is the highest, which is $6.21 \times 10^6 \Omega\text{m}$, and CB has the lowest resistivity, which is $9.31 \times 10^2 \Omega\text{m}$. Carbon black is electrically conductive; most carbon black grades have an electrical volume resistivity

in the range of 10–10000 Ωm (Spahr et al., 2017). For conductivity, CB has the highest conductivity, $1.07 \times 10^{-3} \Omega\text{m}$, while CoF has the lowest conductivity, $1.61 \times 10^{-8} \Omega\text{m}$. The resistivity of CoFCB nanocomposite is drastically lowered compared to the pure CoF sample. This result might be due to the presence of carbon black in the nanocomposite and the opposite for the conductivity values. As the content of carbon black in CoFCB nanocomposite increases, the resistivity decreases while the conductivity increases. The resistivity of all samples is in the range of resistivity of semiconductor materials, $10^{-6} - 10^6 \Omega\text{m}$, which concludes that all samples are semiconductor materials.

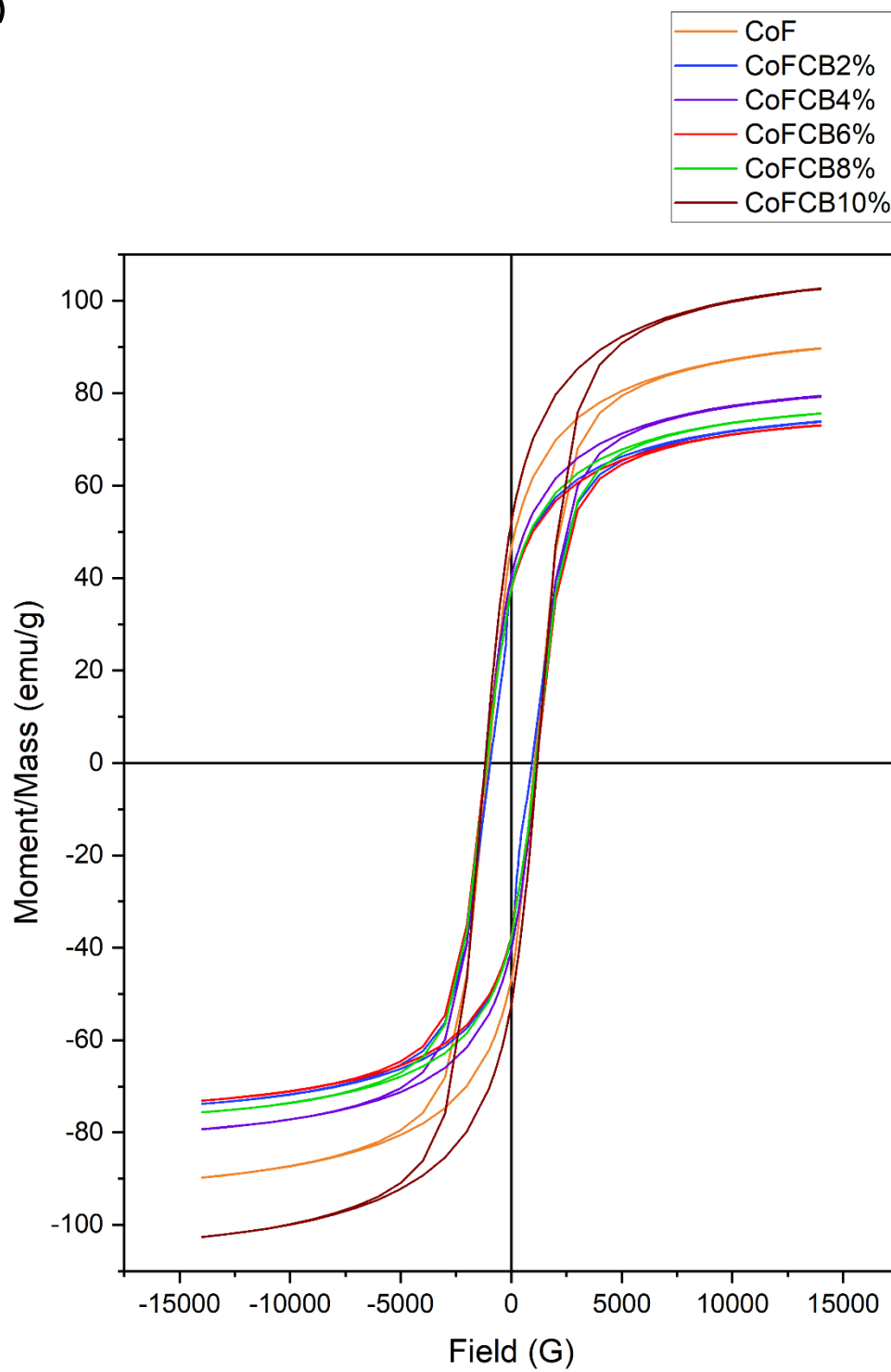
Table 4.2: Resistivity and Conductivity Values of CoF, CB and CoFCB nanocomposites

Sample	Resistivity/ ρ (Ωm)	Conductivity (S/m)
CoF	6.21×10^6	1.61×10^{-8}
CoFCB2%	7.95×10^4	1.26×10^{-5}
CoFCB4%	1.22×10^4	8.18×10^{-5}
CoFCB6%	6.65×10^3	1.50×10^{-4}
CoFCB8%	3.34×10^3	3.00×10^{-4}
CoFCB10%	4.32×10^3	2.31×10^{-4}
CB	9.31×10^2	1.07×10^{-3}

4.5 Magnetic Properties

The magnetic properties of cobalt ferrite, carbon black and cobalt ferrite/carbon black nanocomposite were measured using a vibrating sample magnetometer. Figure 4.7 shows the hysteresis loops for all samples. From Figure 4.7 (a), the large loops for CoF and CoFCB nanocomposites indicate the magnetically hard nature of the ferrite nanoparticles where they have huge coercivity. From Figure 4.7(b), the hysteresis loop of CB is the smallest due to its non-magnetic characteristic (diamagnetic). There is inconsistency in the size of the hysteresis loop as the carbon black content increases. The size of CoFCB from the weight percentage of 2%-8% is smaller than the pure CoF, while the size of CoFCB10% is bigger than the pure CoF and is the biggest hysteresis loop among the samples. From the hysteresis loop graph, the increasing size of the loop indicates increasing saturation magnetization, retentivity and coercivity of the sample.

(a)



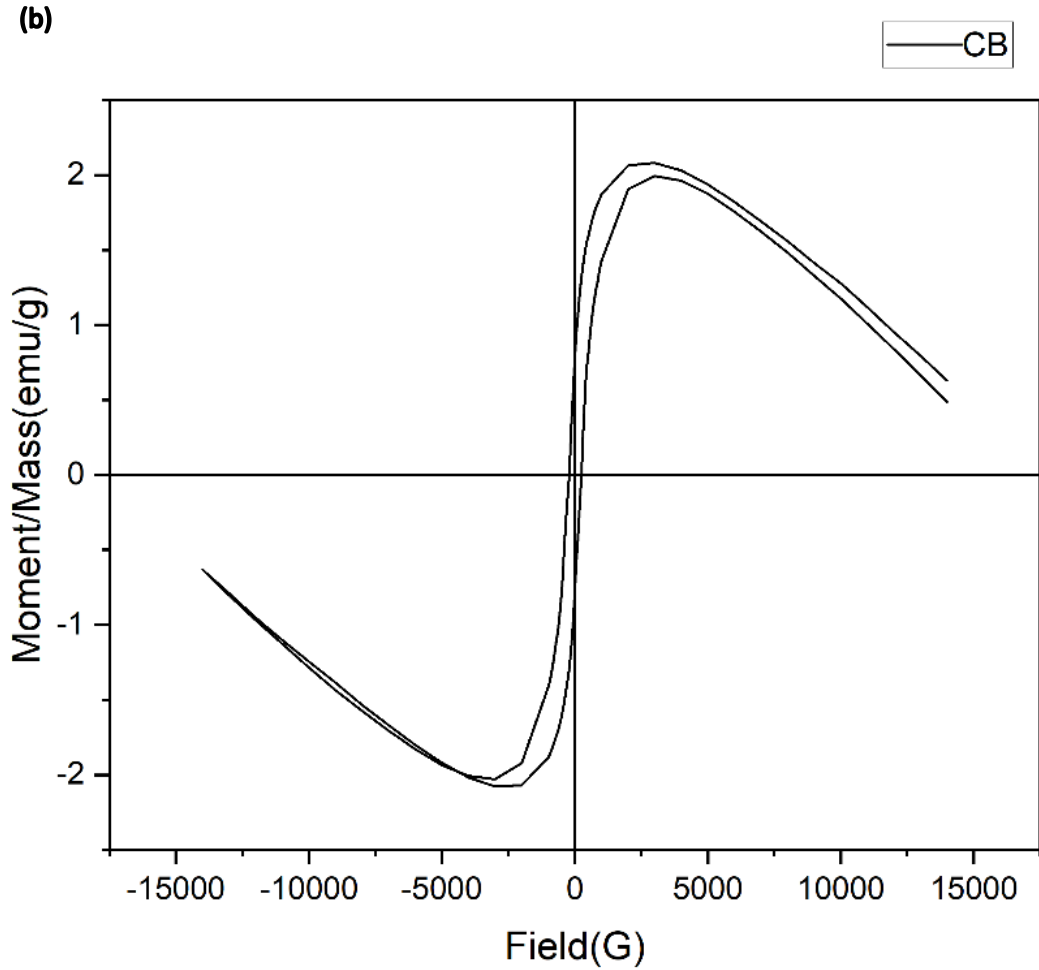


Figure 4.7: Magnetic hysteresis loops for (a) CoF and CoFCB nanocomposites (b) CB

The saturation magnetization (M_s), retentivity (M_r) and coercivity (H_c) are gained from the hysteresis loop and shown in Table 4.3. From Table 4.3, there is also inconsistency on the value of saturation magnetization, retentivity and coercivity by increasing the content of carbon black in the nanocomposite. The saturation magnetization for CoF is 89.768 emu/g, while CB is 2.0797 emu/g. For CoFCB2% to CoFCB10% nanocomposite, the value of saturation magnetization is between 73.083 emu/g to 102.65 emu/g. From Table 4.3, the value of saturation magnetization of CoFCB nanocomposites is smaller than pure CoF except for CoFCB10%, which has the highest saturation magnetization among all samples. The decreases in saturation

magnetization are due to the reduction of magnetic ferrite, which causes the decreases in the net magnetic moment of the composite (Rashid et al., 2013). The highest value of saturation magnetization of CoFCB10% might be due to the increase of the average crystallite size plus improvement in crystallinity, both working to strengthen the long-range magnetic ordering (Stein et al., 2018). For the retentivity of nanocomposites, the value ranges from 37.56 emu/g to 52.229 emu/g. The coercivity values of nanocomposites vary between 944.83 G to 1179.7 G. The increases in the coercivity values as the carbon black content increases may be due to increases the surface spin disorder of the investigated system (Ateia et al., 2019). Appendix D shows the hysteresis loop of each sample.

Table 4.3: Saturation Magnetization, Retentivity and Coercivity Values of cobalt ferrite, carbon black, and cobalt ferrite/carbon black Nanocomposites

Sample	Saturation Magnetization, M_s (emu/g)	Retentivity, M_r (emu/g)	Coercivity, H_c (G)
CoF	89.77	46.71	1044.50
CoFCB2%	73.85	38.17	944.83
CoFCB4%	79.36	40.32	1138.20
CoFCB6%	73.08	37.56	1149.80
CoFCB8%	75.62	37.80	1117.70
CoFCB10%	102.65	52.23	1179.70
CB	2.08	0.78	233.73

CHAPTER 5

CONCLUSION AND SUGGESTION

5.1 Conclusion

In conclusion, the methodology of synthesizing cobalt ferrite nanoparticles using the co-precipitation method and the preparation of cobalt ferrite/carbon black nanocomposites by varying the content of carbon black at a weight percentage of 2%, 4%, 6%, 8% and 10% is successfully discussed. Besides that, the secondary data which is the data from the previous final year student of cobalt ferrite, carbon black, and cobalt ferrite/carbon black nanocomposites is successfully characterized by X-Ray diffraction (XRD), Field Emission Scanning Electron Microscope (FESEM). 2 point-probe, Ultraviolet-visible (UV-Vis) Spectrometer and Vibrating Sample Magnetometer (VSM) respectively. The structural, morphological, optical band gap, electrical and magnetic properties of cobalt ferrite, carbon black, and cobalt ferrite/carbon black nanocomposites are successfully analyzed.

From the XRD analysis, cobalt ferrite nanoparticle is successfully synthesized as CoF's XRD pattern shows peaks of the single-phase spinel cubic structure of ferrite. XRD pattern of CoF has peaks at $2\theta=30.34, 35.70, 37.34, 43.34, 53.72, 57.26, 62.84$ and 74.30 which indicated the planes respectively at (022), (113), (222), (004), (224), (115), (044) and (335). The XRD pattern of CB has peaks at $2\theta= 26.72$ and 43.14 , which correspond with (002). There is no secondary phase showing in the XRD analysis of CoFCB nanocomposite. The intensity of the XRD pattern of CoFCB nanocomposite increases as the weight percentage of CB increases in the nanocomposite.

For morphological properties, the particle size distribution of CoF nanoparticles ranges from 50-110 nm with an average particle size of 73 nm. At the same time, the particle size of carbon black ranges from 40 nm to 100 nm, with an average particle size of 64 nm. From the FESEM micrograph of CoFCB nanocomposites, it is hard to distinguish between cobalt ferrite and carbon black nanoparticles, but from the EDX spectra, it is confirmed that carbon black nanoparticles are presented in CoFCB nanocomposites. As the carbon black content in the CoFCB nanocomposite increases, the peak of C in the EDX spectrum increases, which indicates the increasing carbon black content.

For optical properties, CoF's optical band gap energy is 1.36 eV, while CB's optical band gap energy is 1.01 eV. The band gap energy decreases from 1.24 eV to 1.04 eV due to the increasing carbon black concentration in cobalt ferrite/carbon black nanocomposites.

For electrical properties, the resistivity of CoF nanocomposite is the highest, which is $6.21 \times 10^6 \Omega \cdot \text{m}$, and CB has the lowest resistivity, which is $9.31 \times 10^2 \Omega \cdot \text{m}$. CB has the highest conductivity, $1.07 \times 10^{-3} \Omega \cdot \text{m}$, while CoF has the lowest conductivity, which is $1.61 \times 10^{-8} \Omega \cdot \text{m}$. As the content of carbon black in CoFCB nanocomposite increases, the resistivity decreases while the conductivity increases.

For magnetic properties, the narrow loops for CoF and CoFCB nanocomposites indicate the soft nature of the ferrite nanoparticles where they have small coercivity. The hysteresis loop of CB is the smallest due to its non-magnetic characteristic. The saturation magnetization for CoF is 89.768 emu/g, while CB is 2.0797 emu/g. For CoFCB2% to CoFCB10% nanocomposite, the value of saturation magnetization is between 73.083 emu/g to 102.65 emu/g. For the retentivity of nanocomposites, the

value ranges from 37.56 emu/g to 52.229 emu/g. The values of coercivity of nanocomposites vary between 944.83 G to 1179.7 G.

5.2 Suggestion

This research has successfully analyzed the structural, morphological, optical band gap, electrical and magnetic properties of cobalt ferrite, carbon black, and cobalt ferrite/carbon black nanocomposites. The research has raised new questions and possibilities. Some suggestions that can be made for future research:

1. Synthesize cobalt ferrite by using co-precipitation with varying parameters such as the pH values, sintering temperature, and the duration and rate of stirring.
2. Synthesize cobalt ferrite using other methods such as sol-gel, solvothermal, and hydrothermal.
3. Synthesize cobalt ferrite/carbon black nanocomposite with weight percentages of carbon black such as 10, 12, 14, 16, 18 and 20%.
4. Synthesize cobalt ferrite nanocomposite using co-precipitation method with different carbon materials such as graphite and graphene.

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APPENDICES

APPENDIX A

Preparation of ferrite samples using co-precipitation method

1. **Chemical formula**



2. **Mass of raw material for the preparation of 10 g of cobalt ferrite**

Atomic Weight of Element

Co=58.9332 g/mol

Fe= 55.847 g/mol

N=14.0067 g/mol

Na= 22.98977 g/mol

O=15.9994 g/mol

H=1.00797 g/mol

Calculation for Relative Molecular Mass

$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$

$$= 58.9332 + 14.0067(2) + 15.9994(12) + 1.00797(12)$$

$$= 291.03504 \text{ g/mol}$$

$2\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$

$$= 2(55.847 + 14.0067(3) + 15.9994(18) + 1.00797(10))$$

$$= 807.99952 \text{ g/mol}$$

8NaOH

$$= 22.98977(8) + 15.9994(8) + 1.00797(8)$$

$$= 319.9772 \text{ g/mol}$$

CoFe_2O_4

$$= 58.9332 + 55.847(2) + 15.9994(4)$$

$$= 234.6032 \text{ g/mol}$$

$$\begin{aligned}
 &8\text{Na}(\text{NO}_3) \\
 &= 22.98977(8) + 14.0067(8) + 15.9994(24) \\
 &= 679.95736 \text{ g/mol}
 \end{aligned}$$

$$\begin{aligned}
 &28\text{H}_2\text{O} \\
 &= 1.00797(59) + 5.9994(28) = 504.52952 \text{ g/mol}
 \end{aligned}$$

Desired weight of produced 10g of CoFe_2O_4

$$\frac{\text{Mass of desired ferrite (Total mass of product)}}{\text{Molecular mass of } \text{CoFe}_2\text{O}_4}$$

$$\begin{aligned}
 &= \frac{10 \text{ g (1418.99008 g/mol)}}{234.6032} \\
 &= 60.4847 \text{ g}
 \end{aligned}$$

$$\begin{aligned}
 &\text{Mass of } \text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O} \\
 &\frac{(291.035049 \text{ g/mol})(60.4847 \text{ g})}{1418.99008 \text{ g}} = 12.4054 \text{ g}
 \end{aligned}$$

$$\begin{aligned}
 &\text{Mass of } 2\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O} \\
 &\frac{(807.99952 \text{ g/mol})(60.4847 \text{ g})}{1418.99008 \text{ g}} = 34.4411 \text{ g}
 \end{aligned}$$

$$\begin{aligned}
 &\text{Mass of } 8\text{NaOH} \\
 &\frac{\left(\frac{319.97712 \text{ g}}{\text{mol}}\right)(60.4847 \text{ g})}{1418.99008 \text{ g}} = 13.6391 \text{ g}
 \end{aligned}$$

3. Volume of each element to produce CoFe_2O_4

$$\text{mole}(n) = \frac{\text{mass of solute}}{\text{molar mass}}$$

$$\text{Molarity} = \frac{\text{mol}}{\text{volume}}$$

Molar mass = mass of 1 mole of solute

Volume of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$

$$\text{mole}(n) = \frac{12.4054 \text{ g}}{291.03504 \text{ g/mol}} = 0.04263 \text{ mol}$$

$$\text{Molarity} = \frac{0.04263 \text{ mol}}{0.1 \text{ L}}$$

Volume = 100 ml

Volume of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$

$$\text{mole}(n) = \frac{34.4411 \text{ g}}{403.9998 \text{ g/mol}} = 0.08525 \text{ mol}$$

$$\text{Molarity} = \frac{0.08525 \text{ mol}}{0.1 \text{ L}} = 0.8525$$

Volume = 100 ml

Volume of NaOH

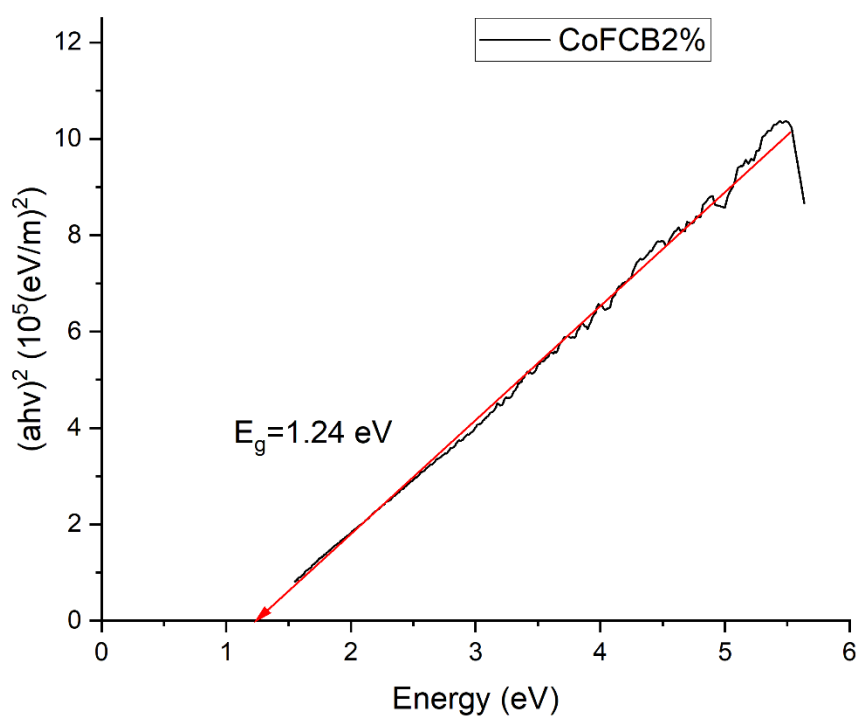
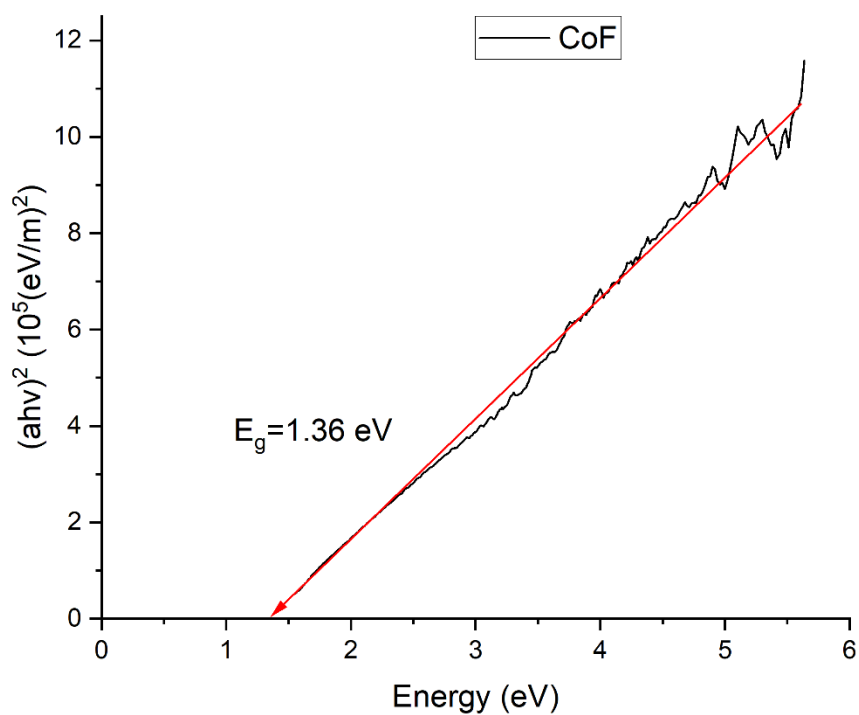
$$\text{mole}(n) = \frac{13.6391 \text{ g}}{39.9972 \text{ g/mol}} = 0.3410 \text{ mol}$$

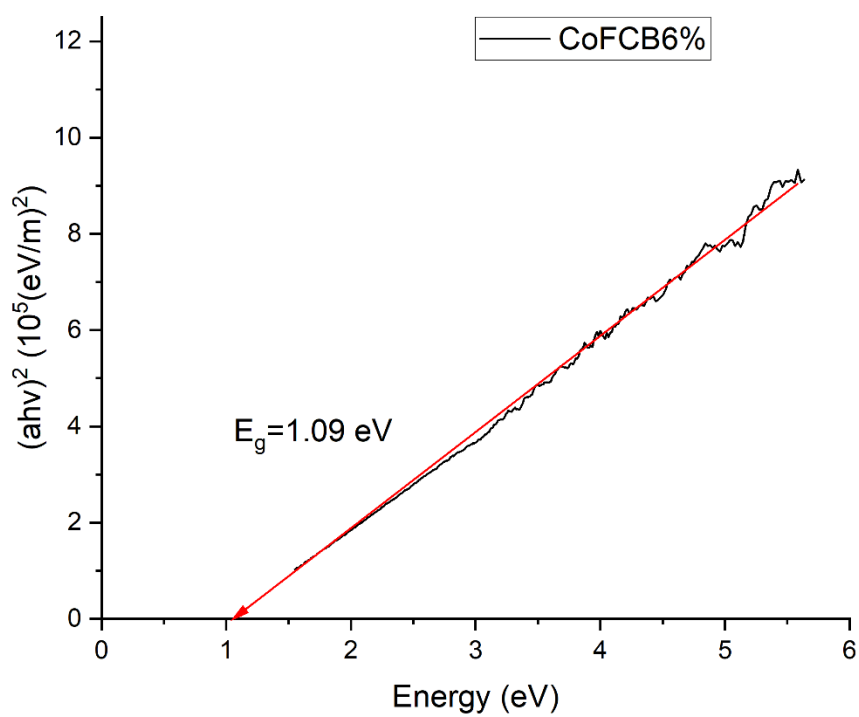
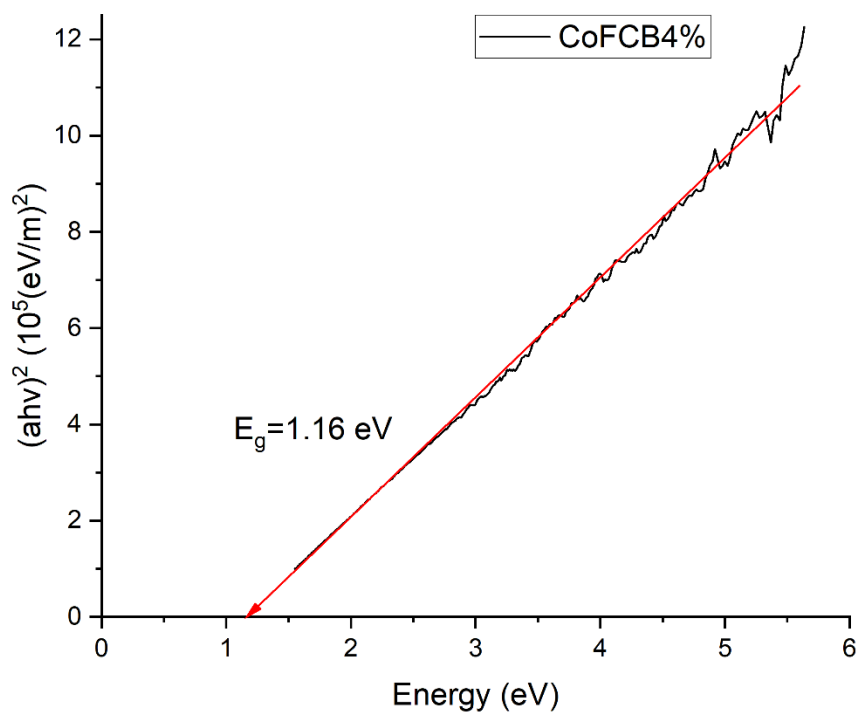
$$\text{Molarity} = \frac{0.3410 \text{ mol}}{0.4 \text{ L}} = 0.08525$$

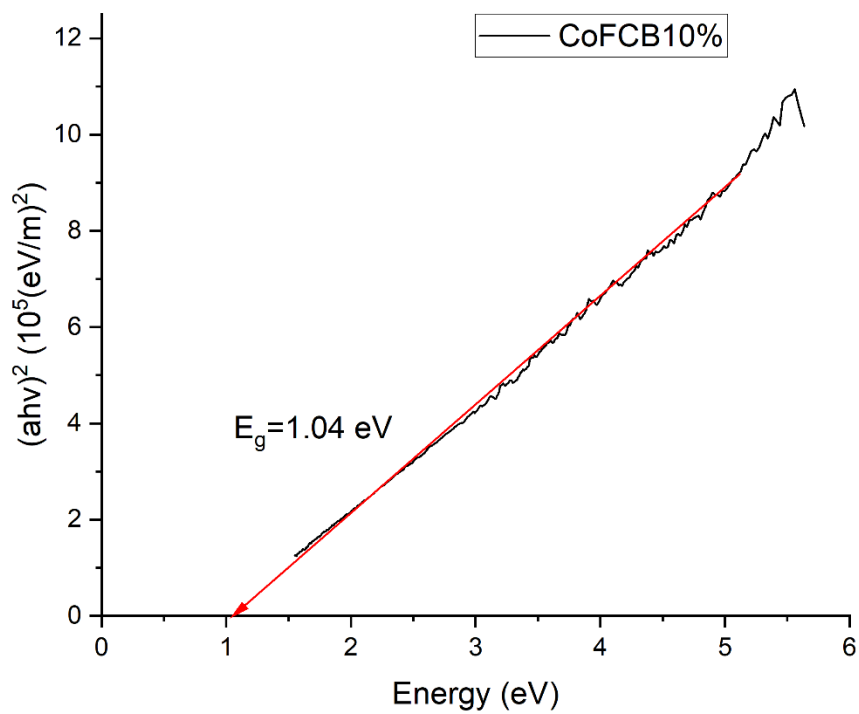
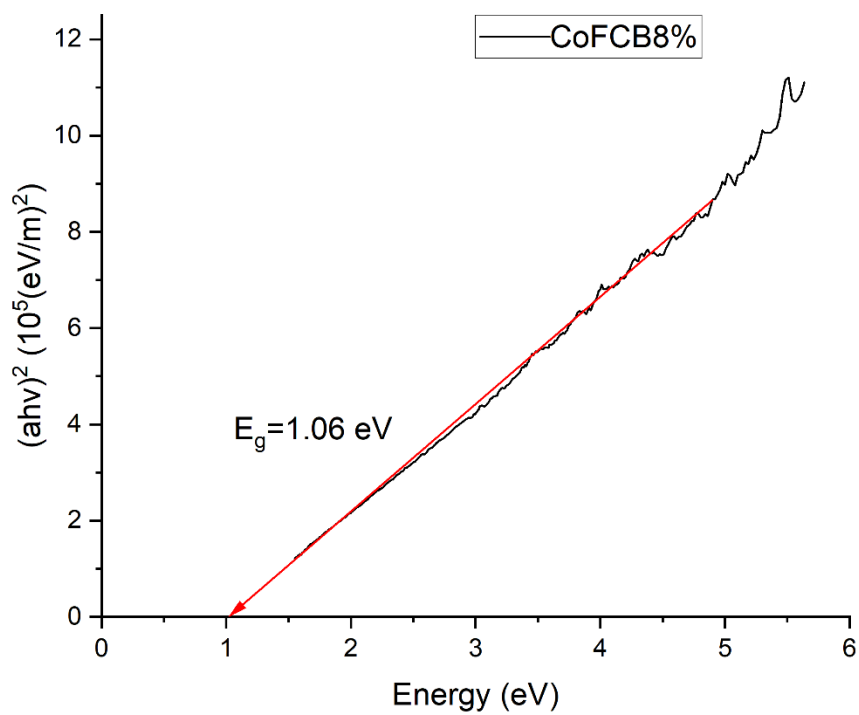
Volume = 400 ml

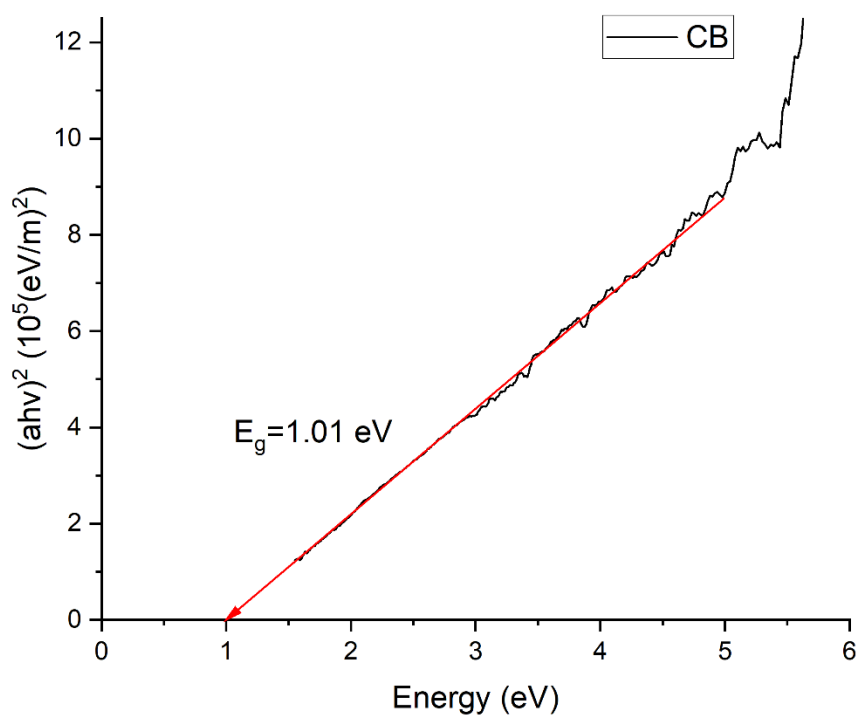
APPENDIX B

Tauc plot graph of the optical band gap energy calculated by extrapolation of the linear part of $(\alpha h\nu)^2$ versus (hc/λ)



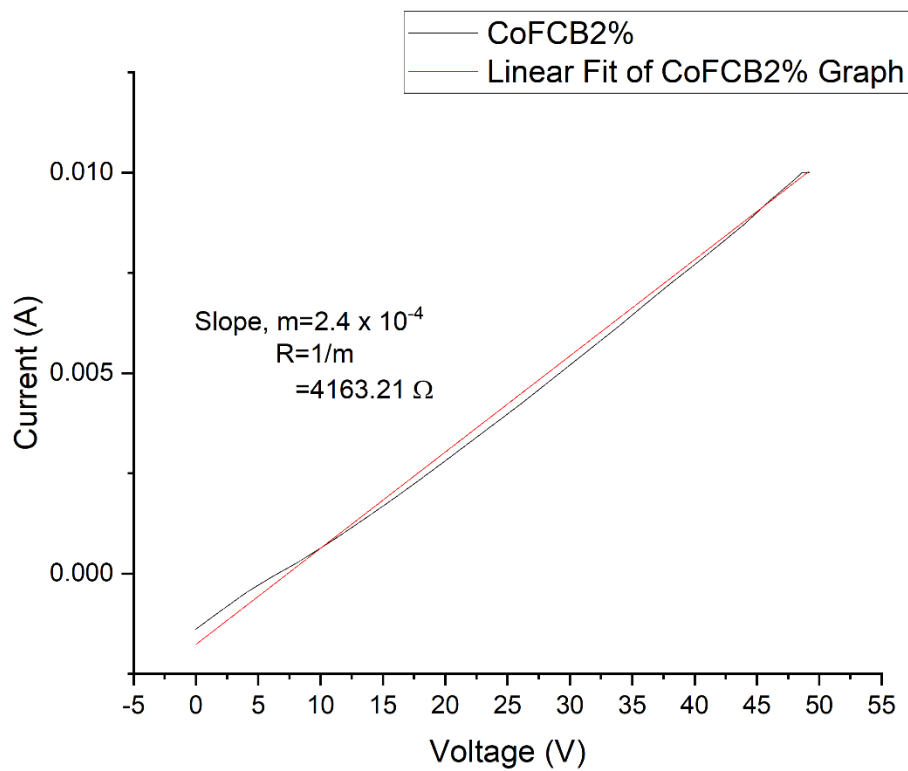
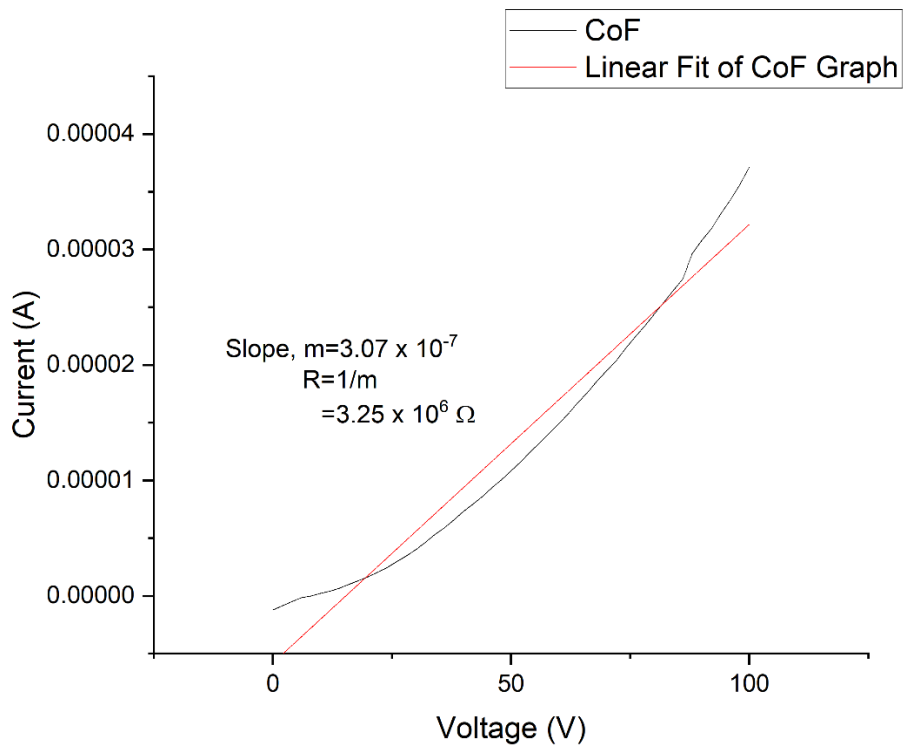


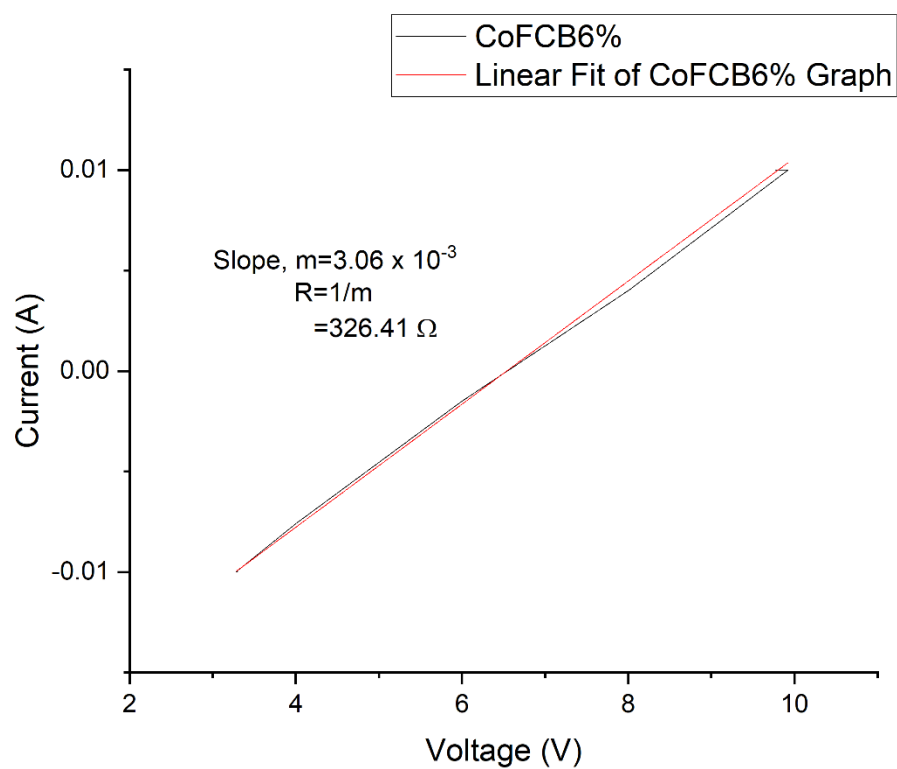
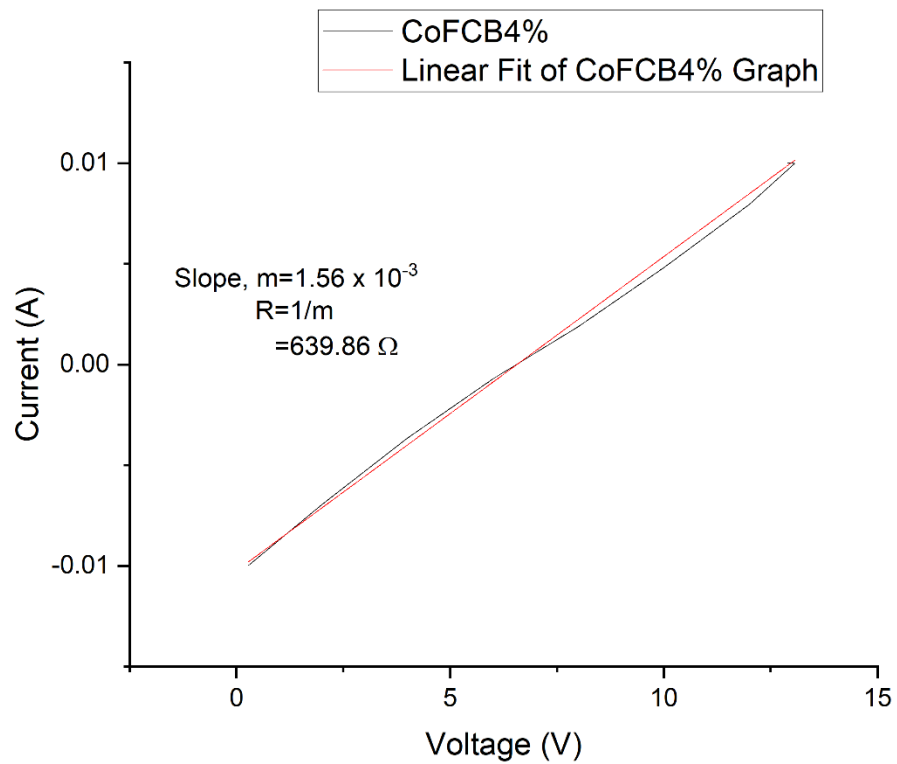


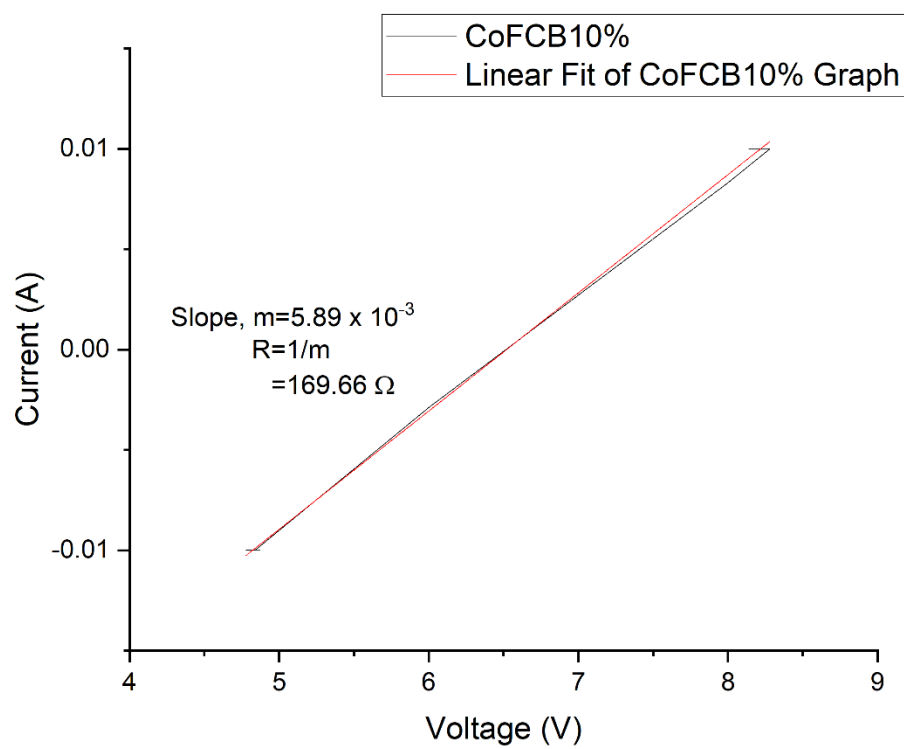
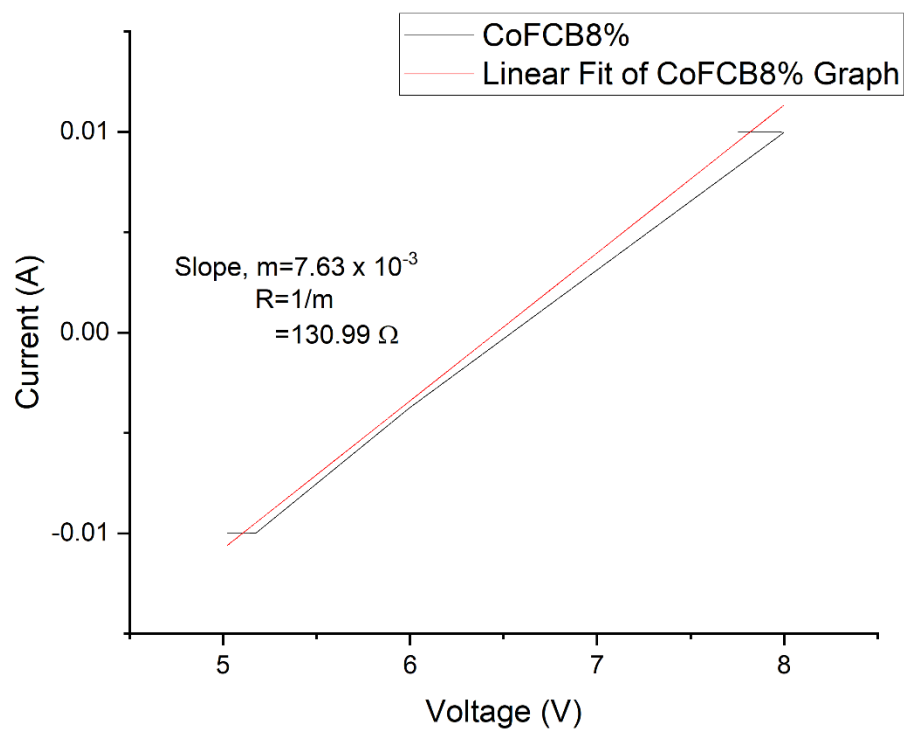


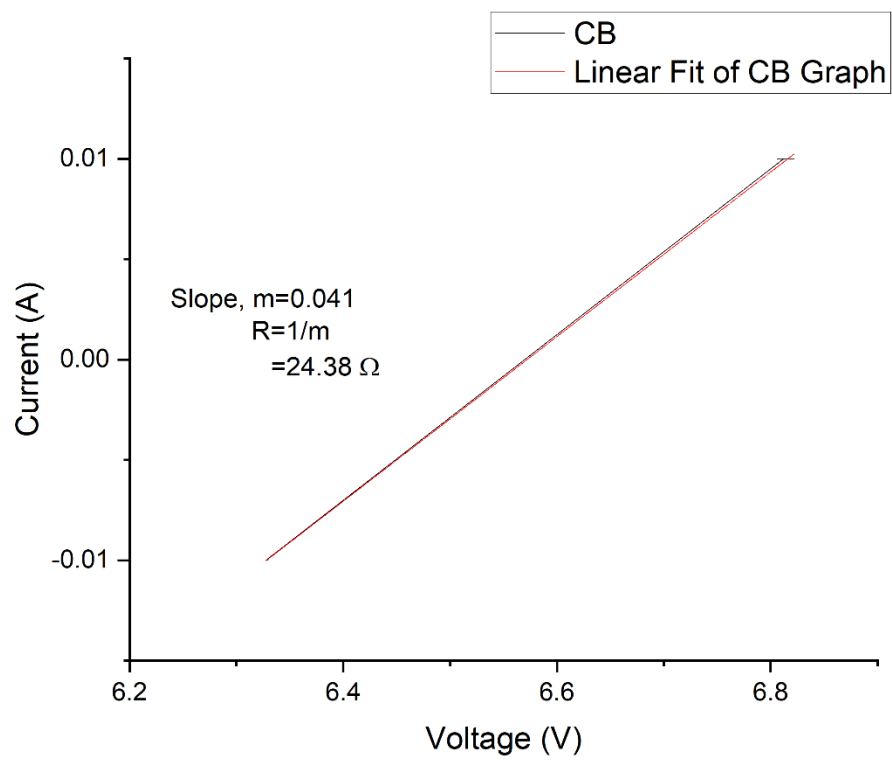
APPENDIX C

Graph of I-V of cobalt ferrite, carbon black cobalt ferrite/carbon black nanocomposite with varying weight percentage of carbon black





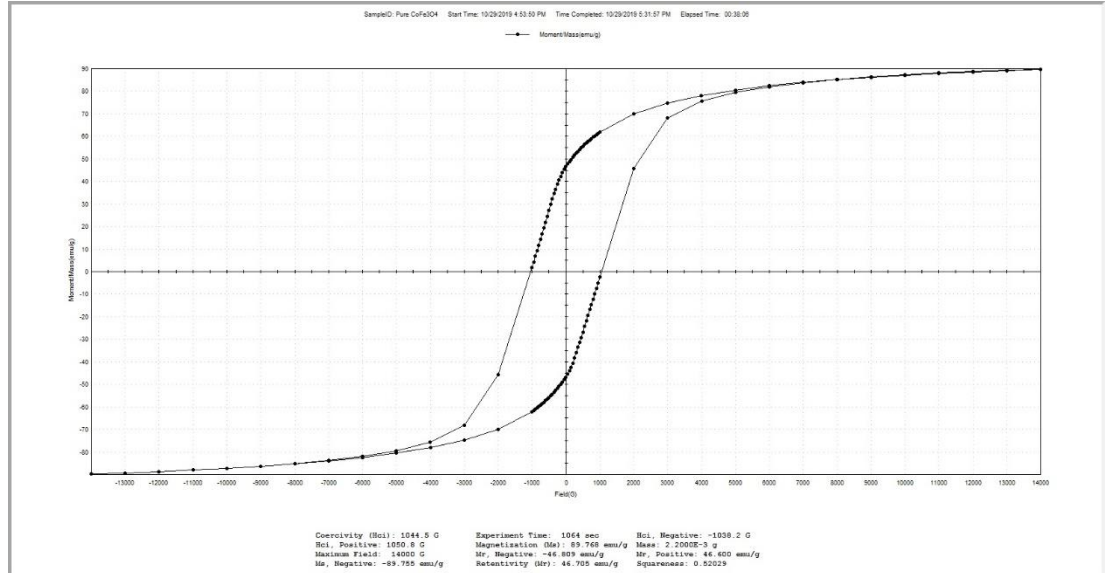




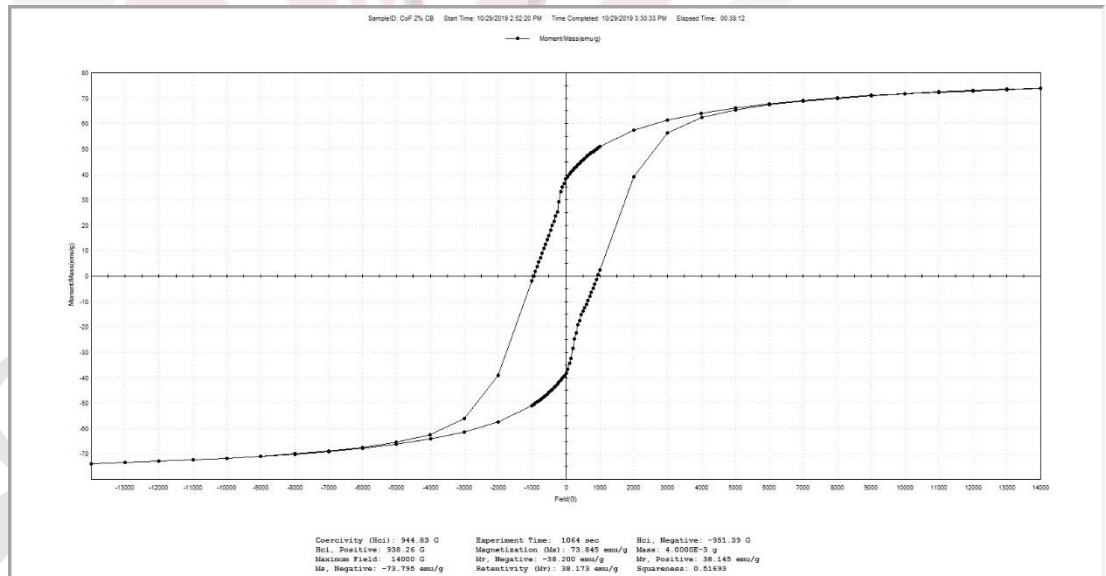
APPENDIX D

Hysteresis loop of cobalt ferrite, carbon black cobalt ferrite/carbon black nanocomposite with varying weight percentage of carbon black

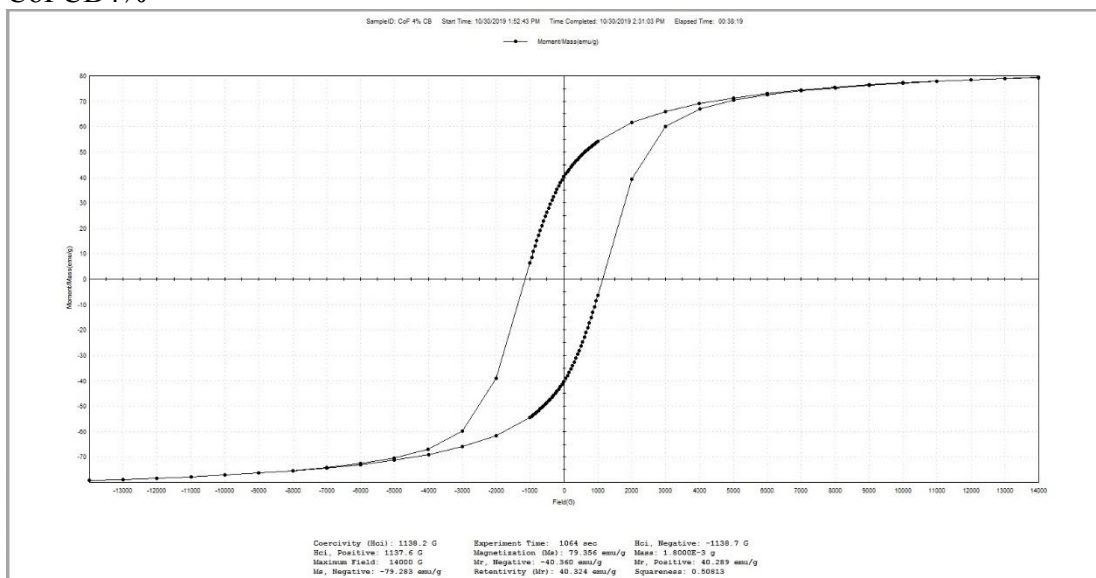
a) Pure CoF



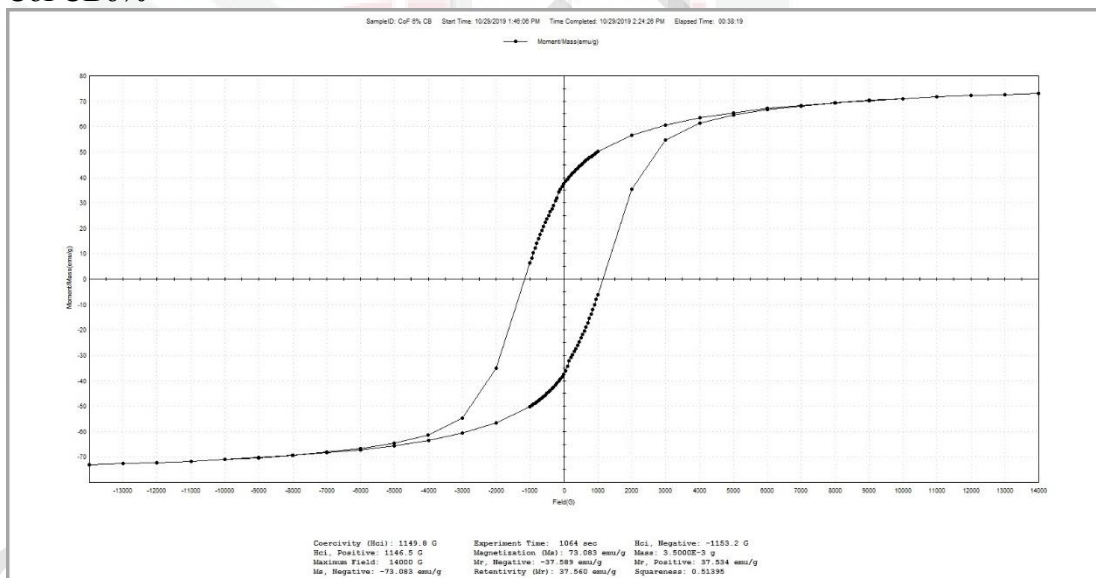
b) CoFCB2%



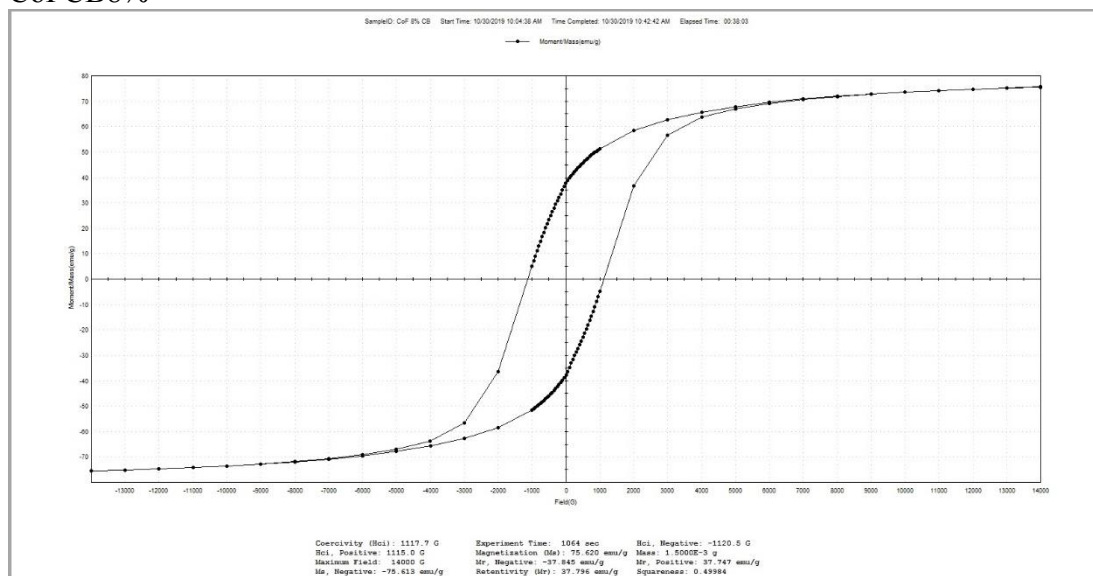
c) CoFCB4%



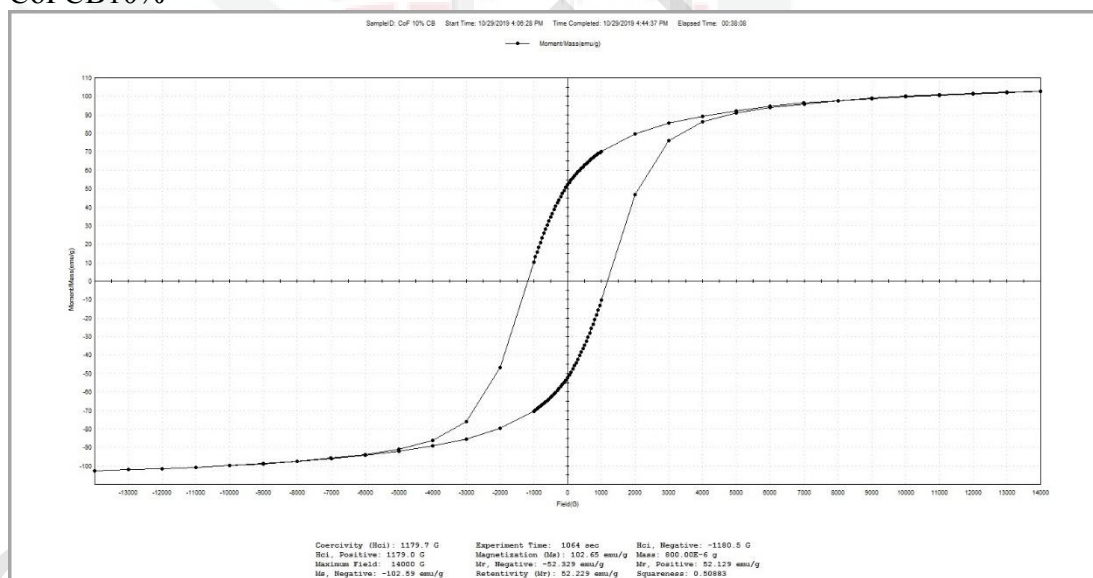
d) CoFCB6%



e) CoFCB8%



f) CoFCB10%



g) CB

