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**Preparation and investigation the Structural and Optical Properties
of (PMMA_Ferrite) Nano-composites**

A research

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By

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بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

{ وَقُلْ رَبِّ زِدْنِي عِلْمًا }

صَدَقَ اللَّهُ الْعَلِيِّ الْعَظِيمِ

سورة يوسف آية

Dedication

To my dear Father

To my beloved Mother

To my Brothers and Sisters

To my Teachers

To my Friends

To everyone who helped me

I dedicate my work

Hamza

Acknowledgment

thanks to Allah, Lord of the Worlds, and blessings and peace be upon our master Muhammad and his good, pure household and his chosen, righteous companions.

And after..

After thanking Allah Almighty for completing this research, I would like to offer my sincere thanks and gratitude to the respected supervisor , Dr. Sameer Hassan Hadi for his suggestion of the research topic and for his valuable advice and guidance.

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Abstract

films were prepared from thermoplastic polymer compound called polymethyl Methacrylate (PMMA) doped with $\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$ nanoparticles at $x=2$ with different weights ratio of (0, 2, 4, 6 and 8)wt % by solution costing technique and using chloroform alcohol as a solvent at room temperature for fifteen minutes.

Optical Microscope was used to find out the distribution of the Nano-Ferrite ($\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$) within the prepared films in homogeneous manner and there are no lumps in them.

Fourier infrared spectroscopy was used to checkup the prepared films there is no chemical reaction between the components of the material from which the films were prepared .

Field Emission Scanning Electron Microscopy (FE-SEM) is used to visualize very small topographic this examination carry out in order to calculate the grain size of Nano ferrite.

The optical and structural properties of the prepared films were studied using a absorption spectrometer (190 - 1100 nm), optical microscope and Fourier infrared spectrometer, The study proved that (absorption coefficient, transmittance, extinction coefficient, refractive index, real and imaginary dielectric constants), which are the optical properties of the thin film under study, their values increase when $\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$ nanoparticles are added, while their energy gap decreased.

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List of Symbols

Symbol	Physical Meanings
Δk	Changes in wave vector
μ	Mobility of charge carriers
μ_i	Electrical dipole moment
A	Absorbance
B	Constant depended on type of material
c	Velocity of light in vacuum
E	Electrical field intensity
ϵ	Complex dielectric constant
ϵ_1	Real part of the dielectric constant
ϵ_2	Imaginary part of the dielectric constant
E_{ph}	Energy of phonon
ϵ_0	Vacuum permittivity
h	Plank constant
$h\nu$	Photon energy
I_A	Absorbed light intensity
I_0	Incident intensity of light
I_p	Conduction current
K	Extinction coefficient
N	Refraction index
n^*	Complex refractive index
N	Concentration of charge carriers
R	Reflectance

T	Temperature
T_r	Transmittance
t_t	Thickness
v	Velocity of light in medium
ν	Frequency
Z	Impedance
α	Absorption coefficient
λ	Wavelength

List of abbreviations

Abbreviations	Meanings
C.B	Conduction band
Mw	Molecular weight
PMMA	Poly(methyl methacrylate)
V.B	Valence band

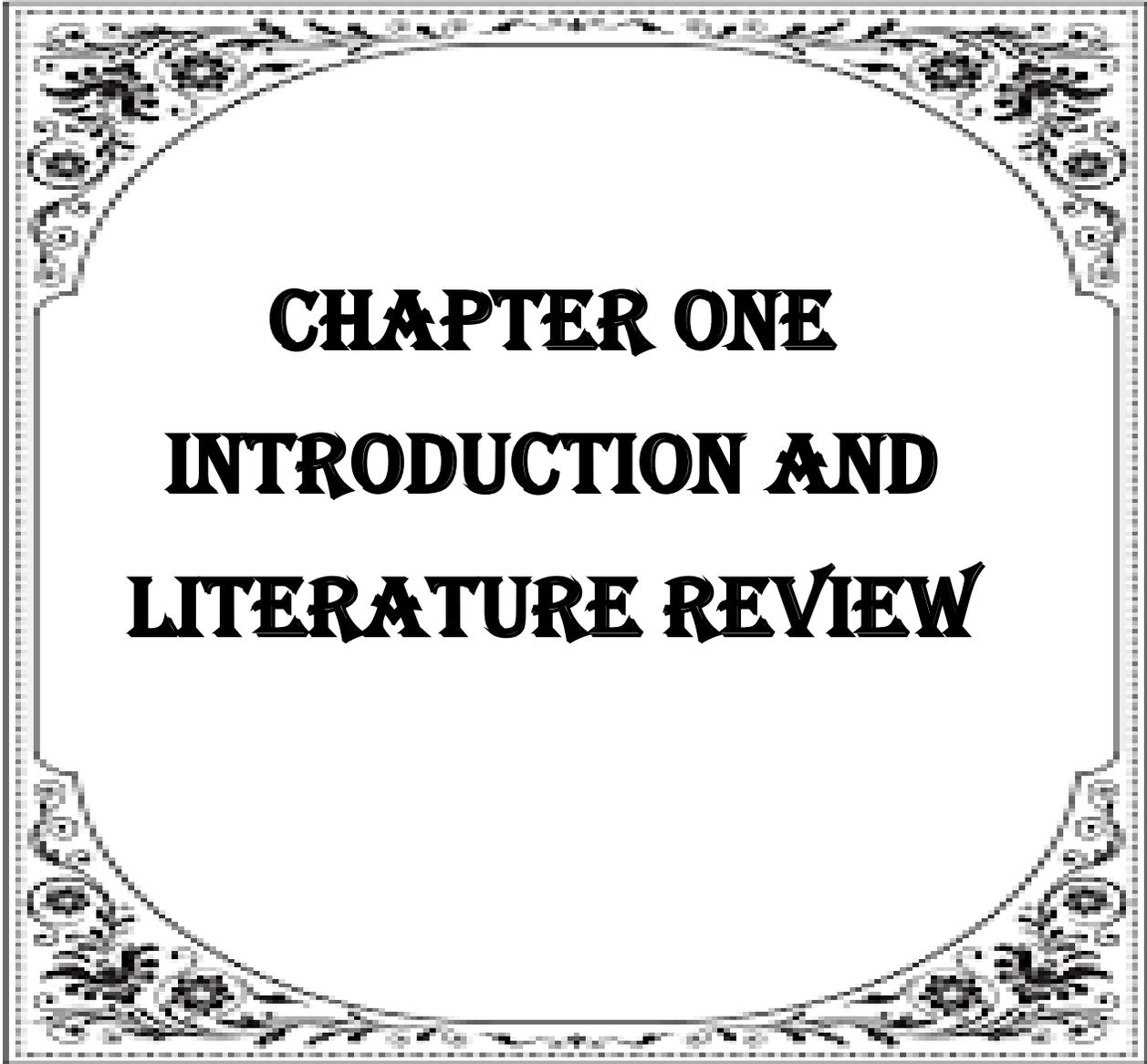
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CHAPTER ONE
INTRODUCTION AND
LITERATURE REVIEW

Chapter one

1-1 Introduction

A polymer (poly "many" + -mer, "part") is a substance or material consisting of very large molecules, or macromolecules[1][2], composed of many repeating subunits[3] Due to their broad spectrum of properties[3] both synthetic and natural polymers play essential and ubiquitous roles in everyday life[4] Polymers range from familiar synthetic plastics such as polystyrene to natural biopolymers such as DNA and proteins that are fundamental to biological structure and function. Polymers, both natural and synthetic, are created via polymerization of many small molecules, known as monomers. Their consequently large molecular mass, relative to small molecule compounds, produces unique physical properties including toughness, high elasticity, viscoelasticity, and a tendency to form amorphous and semicrystalline structures rather than crystals[5] and each unit is called a monomer and their number represents the degree of polymerization linked to each other forming filament polymeric chains as shown in Figure (1 – 1) and may branch out so it is called branched polymer[6] which is a chemical compound that often consists of hydrogen, carbon and other elements[5] Polymers are called in more than one way according to the nature of their formation as simple filamentous polymers such as polystyrene and randomly formed co-polymers resulting from polymerization of more than one monomer such as PMMA-statin and they are also called by trade names such as nylon (polyamides) and PVC (polyvinyl chloride) e.g[1] Sources of polymers are either natural, such as rubber and natural silk, or manufactured such as nylon and plastic[4]

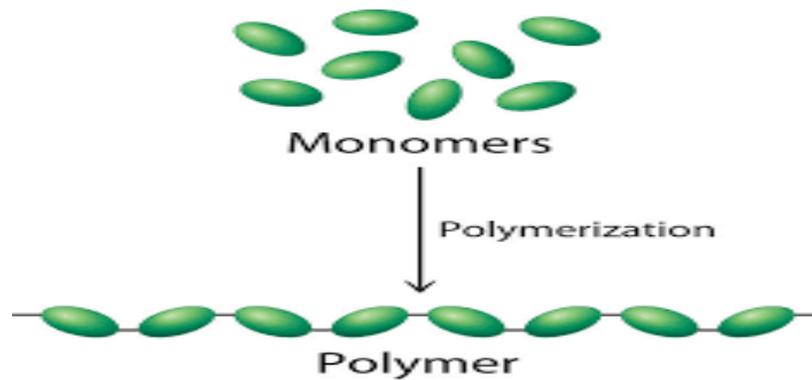


Figure (1-1) Composition of polymers[86]

1-2 Classification of Polymers on The Basis of Technological:

1.2.1 Thermoplastics Polymers:

Solid polymers that are affected by high temperature and become soft and then melt and this property is invested in manufacturing, such as PMMA and polystyrene[8]

1.2.2 Thermosetting Polymers:

Polymers that do not melt by heating and in which chemical changes occur when exposed to pressure and high temperature and do not return to their first state[9] therefore they are not amenable to melting and are used as heat insulating materials such as phenol formaldehyde PF and Epoxy Resin[10]

1.2.3 Elastomers Polymers:

Flexible polymers have ductility due to the low degree of glass transition like rubber[11]

1-3 Physical Properties of Polymers:

To study the physical and mechanical properties of polymers is of great importance in practical applications,[12] where improvements are made and defects in them are eliminated by analyzing these properties such as transmittance, transparency and on their properties they are classified into crystalline, semi-amorphous, amorphous or glass polymers[13] among the physical properties of polymers are:

1-Polymer Melting Point: Polymers are characterized by not having a fixed melting point as they melt in a range of temperatures and depend on the polymer composition, heating speed, molecular weight, copolymerization ... etc. It is one of the important physical constants due to the large changes in the viscosity of the polymer and its specific size[6]

2- Molecular Forces : influential forces between molecules and the sum of these intermolecular forces give certain physical characteristics of the compound, where some polymer solutions do not precipitate at room temperature because of these ionic forces and the polymer's ability to melt or form thin and durable sheets for industrial purposes depends on the strength of the molecules cohesion with each other[14]

3- Molecular Weight : It is the ratio of the total weight of the polymer to the total number of polymeric molecules. The molecular weight of polymers differs from the molecular weight of organic or inorganic compounds due to heterogeneous polymer chains of different lengths, so the average molecular weights is calculated and calculated from the equation[15]

$$M_w = W / \sum N_i \quad (1 - 1)$$

1-4 Nanomaterials:

They are materials of very small size, ranging from (1 - 100) nanometers. The term nanocomposites is used to express the addition of nanoparticles to other materials, especially polymeric compounds, to manufacture new materials with distinctive physical properties compared to pure materials that can be used in many practical applications [16] [17]

1-5 The Importance of Nanomaterials:

Some materials acquire unique properties after adding nanoparticles to them, and an improvement in their functions has been observed (visible, magnetic, and electrical). The studies presented in the search for nanocomposites show properties and advantages that differ from regular compounds that do not contain nanoparticles in their composition [18][19]

1-6 Classification of Nanomaterials:

1-zero-dimensional materials: The materials which are within the nanoscale dimensions smaller than (100 nm) such as transistors and solar cells, which have many applications [20].

2-One-dimensional nanomaterials: they are materials that have only one nanoscale, such as silicon wafers in solar cells or thin films used in packaging [21].

3- Two-dimensional materials: Materials characterized by two nanoscale dimensions, such as nanowires or carbon tubes, have a high electrical conduction capacity, so they are used in solar cells and sensors [22].

4- Three-dimensional nanomaterials: They are materials that have three nanoscale dimensions and include metal powders such as iron oxides used in medical devices as in the subject of the research under study[23]

1-7 A composite material

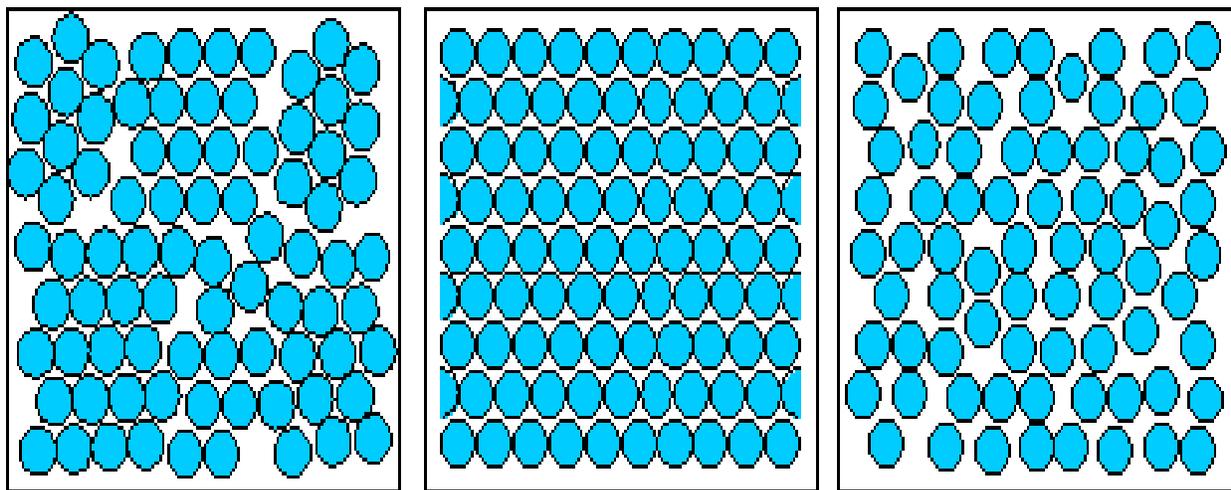
Also called a composition material or shortened to composite, which is the common name) is a material which is produced from two or more constituent materials. These constituent materials have notably dissimilar chemical or physical properties and are merged to create a material with properties unlike the individual elements. Within the finished structure, the individual elements remain separate and distinct, distinguishing composites from mixtures and solid solutions. Typical engineered composite materials include[24]

- ❖ Reinforced concrete and masonry
- ❖ Composite wood such as plywood
- ❖ Reinforced plastics, such as fibre-reinforced polymer or fiberglass
- ❖ Ceramic matrix composites (composite ceramic and metal matrices)
- ❖ Metal matrix composites
- ❖ and other advanced composite materials
- ❖ There are various reasons where new material can be favoured. Typical examples include materials which are less expensive, lighter, stronger or more durable when compared with common materials. More recently researchers have also begun to actively include sensing, actuation, computation and communication into composites,[25] which are known as robotic materials. Composite materials are generally used for buildings, bridges, and structures such as boat hulls, swimming pool panels, racing car bodies, shower stalls, bathtubs, storage tanks, imitation granite and cultured marble sinks and

countertops They are also being increasingly used in general automotive applications[26-28]

1-8 Composition of Nanomaterials:

Several of materials can be manufactured with nanotechnology from a single chemical element such as carbon nanotubes, and they can be manufactured from a chemical compound such as iron oxide[29] As for their atomic arrangement, they may be amorphous substances, as they are random[30] It does not contain a specific crystal arrangement or it may be monocrystalline possessing a specific crystal structure that repeats continuously [31] Figure (1-2) shows this below



Polycrystalline

Single crystalline

Amorphous

Figure (1-2): Types of nanostructures [32]

1-9 Ferrite

Ferrite is one of magnetic oxide compounds, which comprise iron oxide as a major component. Ferrites are usually ferrimagnetic ceramic compounds derived

from iron oxides. Ferrites or ferrimagnetic oxides are dark brown or gray in appearance and very hard and brittle in physical character [33]. Ferrites show dielectric properties, that dielectric property means that even though electromagnetic waves can pass during ferrites, they do not easily conduct electricity. This gives them an advantage over iron, nickel and other transition metals that have magnetic properties in many applications because these metals conduct electricity [34].

Ferrite is chemical compounds (ferromagnetic materials) that are not conducive to electrically and that contain oxygen and at least two magnetic ions that contain the chemical formula (AB_2O_4) , (A) and (B) represent different metal cations consisting of different mixtures of iron oxides. Such as hematite (Fe_2O_3) or magnetite (Fe_3O_4) and other added mineral oxides such as CdO, ZnO, MnO, and AlO. Iron oxide (Fe_2O_3) is more probable than other oxides (manganese, cadmium, zinc, nickel, barium, lithium, etc.) . More than it, these low-cost materials are easy to assemble and offer more formability advantages than their metal and amorphous magnetic counterparts [35].

1.10 Types of Ferrites according to Magnetic Properties :

Ferrites can be divided according to its magnetization into two types hard and soft. This classification depends on the ability of ferrite to magnetize or demagnetize it. Soft ferrites are easily magnetized or demagnetized while hard ferrites are hard to magnetize or demagnetize [36]

1.10.1 Soft Ferrites

It is a type of ceramic that can be easily magnetized this indicates that magnetic materials have a low coercive field and high magnetization is required

in many applications, and the hysteresis loop is long and narrow and therefore the energy loss is very low in these magnetic materials[37]. Soft ferrites have certain advantages over other electromagnetic materials including high electrical resistance, eddy current losses over a wide range and high and stable transmittance temperatures. Many examples of Spinel ferrites like manganese–zinc ferrite $(\text{Mn,Zn,Fe})\text{O}_4$ system are commercially important soft magnets. In addition, lithium ferrite, nickel ferrite, and garnets are other examples of soft ferrites [38].

1.9.1.2 Hard Ferrites:

Hard ferrites of permanent ferrite magnets, which have a high coercivity and high remanence after magnetization, which has the high coercivity that means the materials are very resistant to become demagnetized, an essential properties for a permanent magnet. They also have high magnetic permeability. These so-called ceramic magnets are cheap, and are widely used in house hold products such as refrigerator magnets. Iron oxide and barium or strontium carbonate are used in manufacturing of hard ferrite magnets These are most common hard ferrites:

Strontium ferrite, $\text{SrFe}_{12}\text{O}_{19}$ ($\text{SrO}\cdot 6\text{Fe}_2\text{O}_3$), used in small electric motors, microwave devices, recording media, magneto-optic media, electronic industry and telecommunication [39]

1-10 Literature Survey :

Abdul-Muhsien M at el(2011) [40] prepared the PMMA/ TiO_2 composite with different weight ratios (0,4,6) ,% Wt% of TiO_2 , the study showed improvement optical properties with increasing proportions of TiO_2 in the prepared compounds for a determined range of wavelengths (300-850) nm where it was observed. Increasing the absorption coefficient, real and imaginary dielectric constants, and a

decrease in the energy and transmittance gap of the permissible and prohibited indirect transmission.

Hamed,N.G and Rahen,M, (2014) [41], studied the optical properties for PMMA / Ag compositions prepared by pouring solution with different weight ratios (5, 9, 13) Wt% . The results showed an increase in the absorption coefficient with an increase in the added Ag ratio.

Maryam F Obeys and ,Zaid A Hasan (2018)[42], studied The effect of the magnetic field on improving the optical properties of the doped PMMA polymer with copper nanoparticles caused the increase in the permeability values, the decrease in absorbance, the absorption coefficient, the real and imaginary dielectric constant, the extinction and refraction coefficients.

M .I. Mohammed (2018) [43],discussed the effect of adding ZnO nanoparticles at different concentrations to the PMMA / PVD complex on optical properties. The thin film were prepared using the solution molding method. The results of the examination in the UV-Vis and XRD apparatus showed that direct transition is the dominant one and the light energy gap decreases with increasing the concentration of nanoparticles.

A. Yazgan(2019)[44] studied The effect of addition of Fe₂O₃ and ZnO nanoparticles with different weight ratios on the optical properties was mainly examined Films were prepared using solution casting technology. X-ray diffraction and electron microscopy results showed a decrease in the optical transmittance of thin film with increased anesthesia and dielectric loss at rates higher than pure PMMA.

Ahmed Hashim Hayder M. Abduljalil,(2020) [45] In studied the electronic properties of PMMA doped with ZnO nanoparticles, where theoretically the energy gap, the ion potential, the hardness and the electronegativity were calculated, where the engineering improvement of PMMA was observed and the compatibility with the experimental data and the results PMMA. Use in multiple applications

Zaid A Hasan, Saja F. G Abide(2020)[46] studied the effect of adding nanoparticles-MgO with different weight ratios on the optical properties of the PMMA composite and the effect of the magnetic field on it, as it showed an improvement in the optical properties as the transmittance increased and the absorption values decreased, the absorption, refractive index, and the real and imaginary isolation constant.

Sameer H. Al-nesrawy et. al. (2021) [47], studied polymer blend (PVA-PVP)-Carbon black (C.B N375)nanocomposites.The (PVA-PVP-C.B) nanocomposites are organized by via casting procedure. The optical microscope, FTIR and electrical properties have been studied. The constant of dielectric with the dielectric loss of the samples were reduced with increasing the value of frequency during the application of electric field, while an increasing in the A.C electrical conductivity results existed with the rising the value of the frequency. The electrical conductivity (A.C), dielectric loss and constant of all the samples were increased with the increasing of the carbon black concentrations.

Manar S. and Tom (2021)[48],prepared the (PbO/ PVA-PEG) nanocomposites through adding the different weight concentrations of lead oxide (0,1,3,5,7 wt%). By using casting method. The structural aspects such as optical microscope, FTIR and electrical features of nanocomposites (PVA-CMC/PbO) were examined. The resulting data shows that the dielectric constant decreased along with the decline of

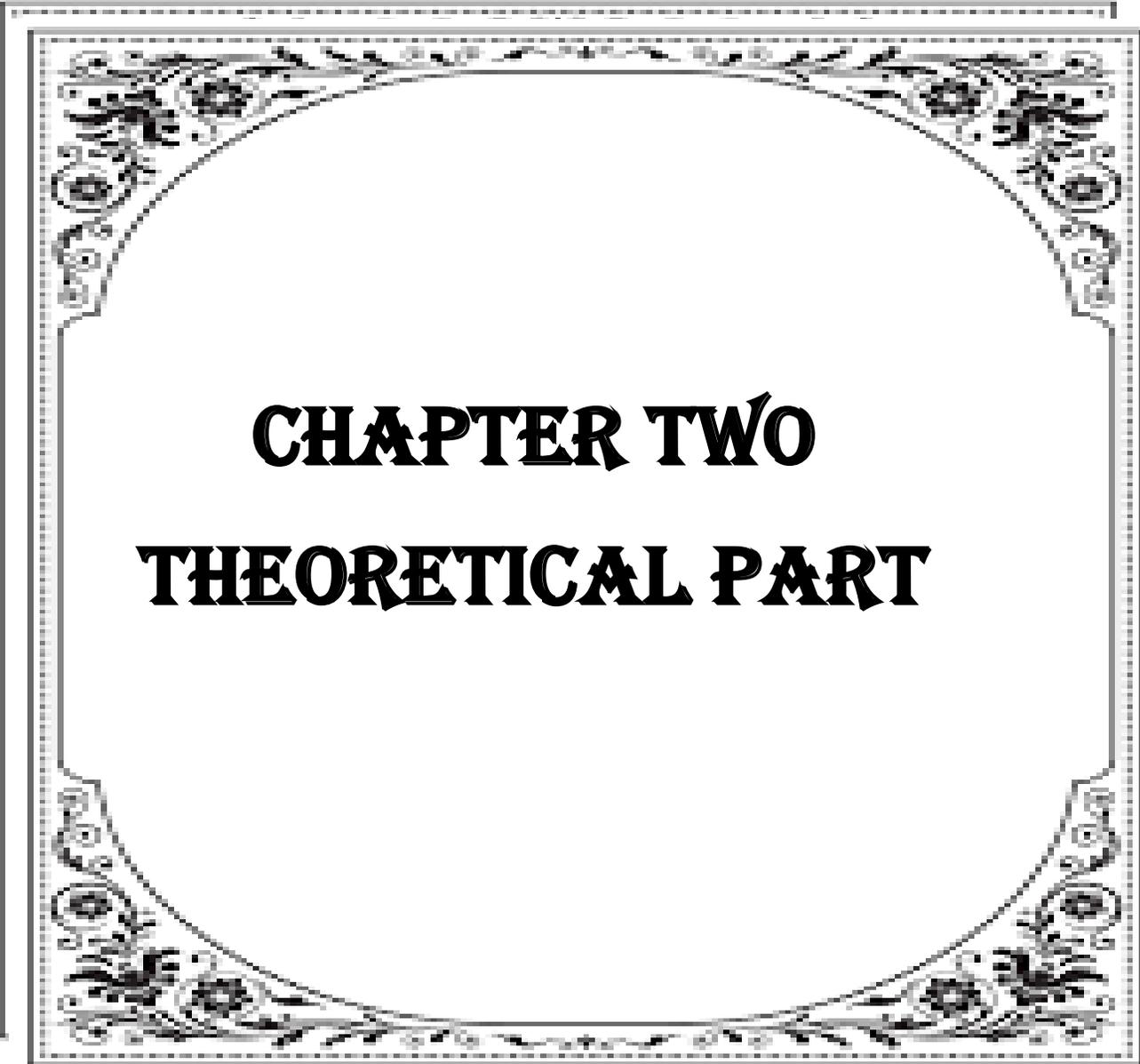
dielectric loss, whereas the frequency value rose while applying of an electric field. As for the electrical conductivity AC, the dielectric loss and dielectric constant of all samples rose along with the increase in lead oxide concentration

Ali Jasim Mohammed (2022) [49], prepared the ferrite $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$ by the co-precipitation method and examined through the XRD-diffraction and confirming the face center cubic spinel phase (FCC) which attributed to the ferrite and found that these materials was Nano-scale. Then with different content of ferrite nanoparticle (1, 3 and 5 wt.%) additive to the (PVP/PVA) polymer matrix to synthesis (PVP/PVA/ferrite) nanocomposite by using casting method and study the Fourier transformation infrared (FTIR), (FE-SEM) and UV-Vis. Spectrophotometer. The FTIR confirming that there is no interactions between (PVP/PVA) polymer matrix and ferrite nanoparticles for all the sample prepared. From FE-SEM, the uniform morphology dispersed of ferrite inside the PVP/PVA blend with spherically shaped nanoparticles and the average grain size increased with increasing of concentration of ferrite. The absorption, absorption coefficient, transmittance and indirect energy band gap has been investigate

1-11 Aims of The Study:

This study aims to:

- Preparation of (PMMA- $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) Nanocomposites films
- Studies several of the Structural and Optical Properties of (PMMA- $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) nanocomposites



CHAPTER TWO
THEORETICAL PART

Chapter Two

2-1 Introduction

This chapter includes a description of the theoretical side of the research topic and all the laws and mathematical equations used to measure the values of the optical physical properties and clarify their physical concepts to interpret the results of the research under study.

2-2 Optical Properties of Polymers:

The optical properties of polymers are of great importance in revealing the type and nature of chemical bonds within the internal structure of the polymer by testing infrared films with the purpose of

determining its use in multiple applications and fields, as well as tests of spectra within the range of ultraviolet rays to find out electronic transitions, the energy gap and the effect of adding nanomaterials to effect changes in optical properties through absorption or emission of a specific wavelength of electromagnetic radiation due to changes in electronic properties with effects of size and quantum[50] [51]

2-2-1 Absorbance:

The absorption is defined as the amount of light absorbed by the film I_A with respect to the amount of light incident on it I_0 , also called optical density and it represents the ratio of the intensity of the light reflected from the sample compared to the intensity of the incident light, which is an amount without units and is calculated according to the equation[52] [53]

$$A = I_A / I_0 \quad (2 - 1)$$

$$A = \log \left(\frac{1}{T_r} \right) \quad (2 - 2)$$

2-2-2 Transmittance:

The transmittance of the film is measured directly, which is the amount of rays reflected from the film to the amount of rays that incident on it according to the equation[54].

$$T = I_T / I_O \quad (2 - 3)$$

2-2-3 Absorption of Coefficient (α):

It is expressed as the loss in the intensity of light upon entering a certain medium [55], and it represents the percentage of the loss in the intensity of light from the light beams incident directly through a given thickness of the material [56] and therefore depends on the amount of absorption and thickness of the thin film according to the following equations

$$dI = -\alpha I dx \quad (2 - 4)$$

When a material of thickness (t_i) is subjected to the intensity of the incident ray (I_o) and the intensity of the transmittance ray I_T , then the integration procedure obtained[57].

$$\int_{I_o}^{I_{T_r}} \frac{dI}{I} = \int_0^{t_i} -\alpha dt \quad (2 - 5)$$

$$\ln I_{T_r} - \ln I_o = -\alpha t_i \quad (2 - 6)$$

$$I_{T_r} = I_o \exp(-\alpha t_i) \quad (2 - 7)$$

$$\frac{I_{T_r}}{I_o} = \exp(-\alpha t_i) \quad (2 - 8)$$

$$T_r = \exp(-\alpha t_i) \quad (2 - 9)$$

$$\frac{1}{T_r} = \exp(\alpha t_i) \quad (2 - 10)$$

$$2.303 \log\left(\frac{1}{T_r}\right) = \alpha t_i \quad (2 - 11)$$

$$A = \log\left(\frac{1}{T_r}\right) \quad (2- 12)$$

$$2.303 \times A = \alpha t_i \quad (2 - 13)$$

$$\alpha = (2.303 \times A) / t_i \quad (2 - 14)$$

2-2-4 Extinction Coefficient (K):

Represents the amount of energy absorbed in the thin film or the amount absorbed by the electrons of the material from the energy of the incident photons, i.e. the extinction or attenuation carrying the electromagnetic wave inside the material. Where it represents the imaginary part of the refractive index given by relationship

$$K = \alpha\lambda / 4\pi \quad (2-15)$$

2-2-5 Refractive Index (n):

It represents the ratio between the speed of light in a vacuum to its speed in the medium, when the grain size changes, the refractive index changes even if the crystal structure is the same [58]

$$n_0 = c / v \quad (2 - 16)$$

c: speed of light in vacuum, v: speed of light in medium

The value of the refractive index is calculated from the equation below:

$$n = \sqrt{\frac{4R}{(R-1)^2} - k_0^2} - \frac{(R+1)}{(R-1)} \quad (2 - 17)$$

The reflectivity can be calculated depending on the law of energy conservation from knowing the absorption and transmission spectra from the following equation[59].

$$R + A + T_r = 1 \quad (2-18)$$

R is the reflectivity, T_r the transmittance, A the absorbance.

2-2-6 Dielectric Constant (ϵ)

Polarization occurs when the incident light rays interact with the charges in the falling medium due to energy absorption in the material, which means that energy loss occurs as a result of this interaction, the polarization of the medium and electronic polarizations are generally common than other polarizations, and the partial properties of the material affect the degree of polarization in addition to the electric field. The complex dielectric constant is obtained by the following equations

$$\epsilon = \epsilon_1 + i \epsilon_2 \quad (2-19)$$

$$\epsilon = (n^*)^2 \quad (2-20)$$

$$(n-ik_0)^2 = \epsilon_1 - i \epsilon_2 \quad (2-21)$$

$$\epsilon = (n^2 - k^2) - i(2nk_0) \quad (2-22)$$

$$\epsilon_1 \text{ real part} = n^2 - k^2 \quad (2-23)$$

$$\epsilon_2 \text{ imaginary part} = 2nk_0 \quad (2-24)$$

The relationship of the dielectric constant to the refractive index is obtained by the equation

$$\epsilon = n^2 \quad (2-25)$$

Note that for the same wavelength, the value of ϵ_1 is greater than ϵ_2 for the films prepared by casting method in this study.

2-3 Absorption Regions

Fundamental Absorption Edge

The basic absorption edge is described as the rapid increase in the absorption rate of the electromagnetic radiation at the moment when the energy gap is equal to the energy of the absorbed radiation. The so-called basic absorption edge occurs and it represents the smallest energy difference between the highest point in the valance band and the lowest point in the conduction band[60]

A-High Absorption Region

This region is shown in Figure (2-1) this area where the absorption coefficient(α) is larger or equal to 10^4 cm^{-1} , and in which the energy gap E_g can be identified and the absorption coefficient is calculated by the following equation: [61]

$$\alpha h \nu = B(h \nu - E_g)^r \quad (2-26)$$

whereas:

B : is a constant that depends on the nature of the substance.

$h \nu$: photon energy in units (eV)

E_g : the energy gap.

r : an exponential parameter that depends on the nature of the transition.

B-Exponential Region :

In Figure (2-1) Part B shows this region where the absorption edge increases exponentially due to the increase in absorption, where the absorption coefficient values are between $(1 < \alpha < 10^4) \text{ cm}^{-1}$ and electronic transitions occur in it from the local levels at the top of the valance band to the extended levels in the conduction band[62]

The absorption factor is calculated from the equation[64]

$$\alpha = \alpha_0 \exp (h \nu / \epsilon_u) \quad (2-27)$$

whereas :

α_0 : the proportionality constant.

ϵ_u : The width of the tails for the positional levels in the energy gap region (the energy of the tails)

C-Low Absorption Region

In Figure (2-1) Part C illustrates this region where the absorption coefficient (α) has a very small value. It is about $\alpha < 1 \text{ cm}^{-1}$ the transition in this region occurs due to the density of the state within the space motion caused by structural errors[63]

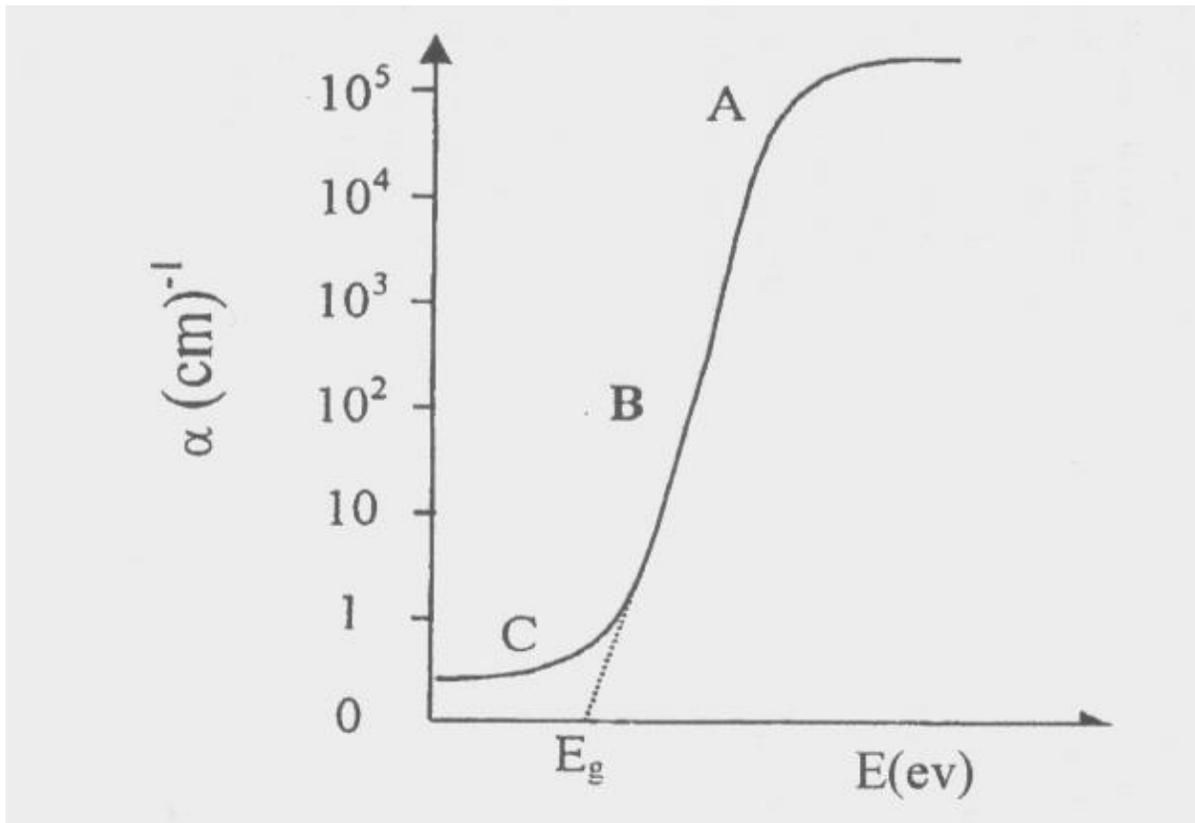


Figure (2-1) : Absorption edge variations with varying the areas of absorption[64]

2-4 The Electronic Transitions :

There are two basic types of electronic transitions [65]

2-4-1-direct electronic transitions:

In general, direct electronic transfers are not accompanied by a perceptible change in electron momentum, and they are of two types[66] Direct electronic transport is permitted, and the permissible electronic transport is done by the movement of electrons in the bounded region between the valence band crest and the watt point in the conduction band,[67] where the value of $r = 1/2$ is achieved and the absorption at $E_g = h \nu$ direct electronic forbidden transition occurs at the neighboring points between the top of the valence band and the bottom of the conduction band, where the value of $r = 3/2$, and the absorption coefficient in this region is calculated from the equation [68]

$$\alpha h \nu = B_0 (h \nu - E_g)^r. \quad (2-28)$$

B_0 is a constant that depends on the nature of the material.

$h \nu$ The energy of a photon is in units of eV.

E_g is the power gap in units of(eV)

r is an exponential parameter that takes the values $(3 / 2 , 2 / 3 , 1 / 2)$ depending on the type of material and the type of electronic transition

2-Indirect electronic transition:

This type of indirect transmission is characterized by the conservation of momentum due to the change in the electron wave vector and is divided into two types[69]

The permissible indirect transition occurs as a result of the electron transfer between the valence band apex and the conduction band bottom at different points of space(K) [72]

2-4-2 The transmission is indirect, prohibited, and it occurs as a result of the transfer of the photon between adjacent points at the top of the valence band and the bottom of the conduction band, as shown in fig (2 - 2) [73]

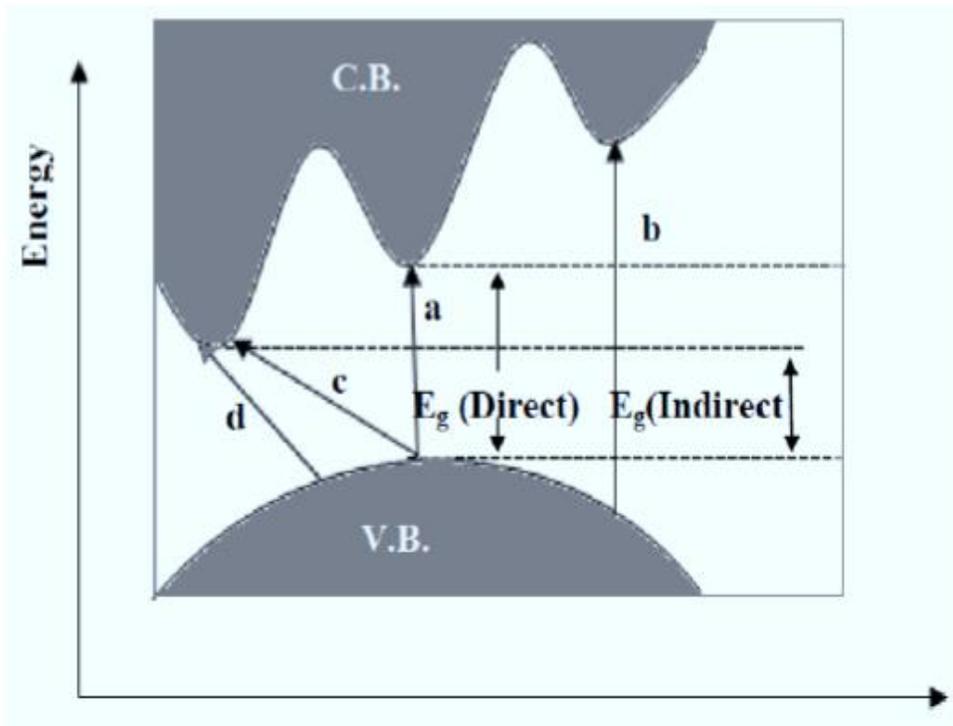
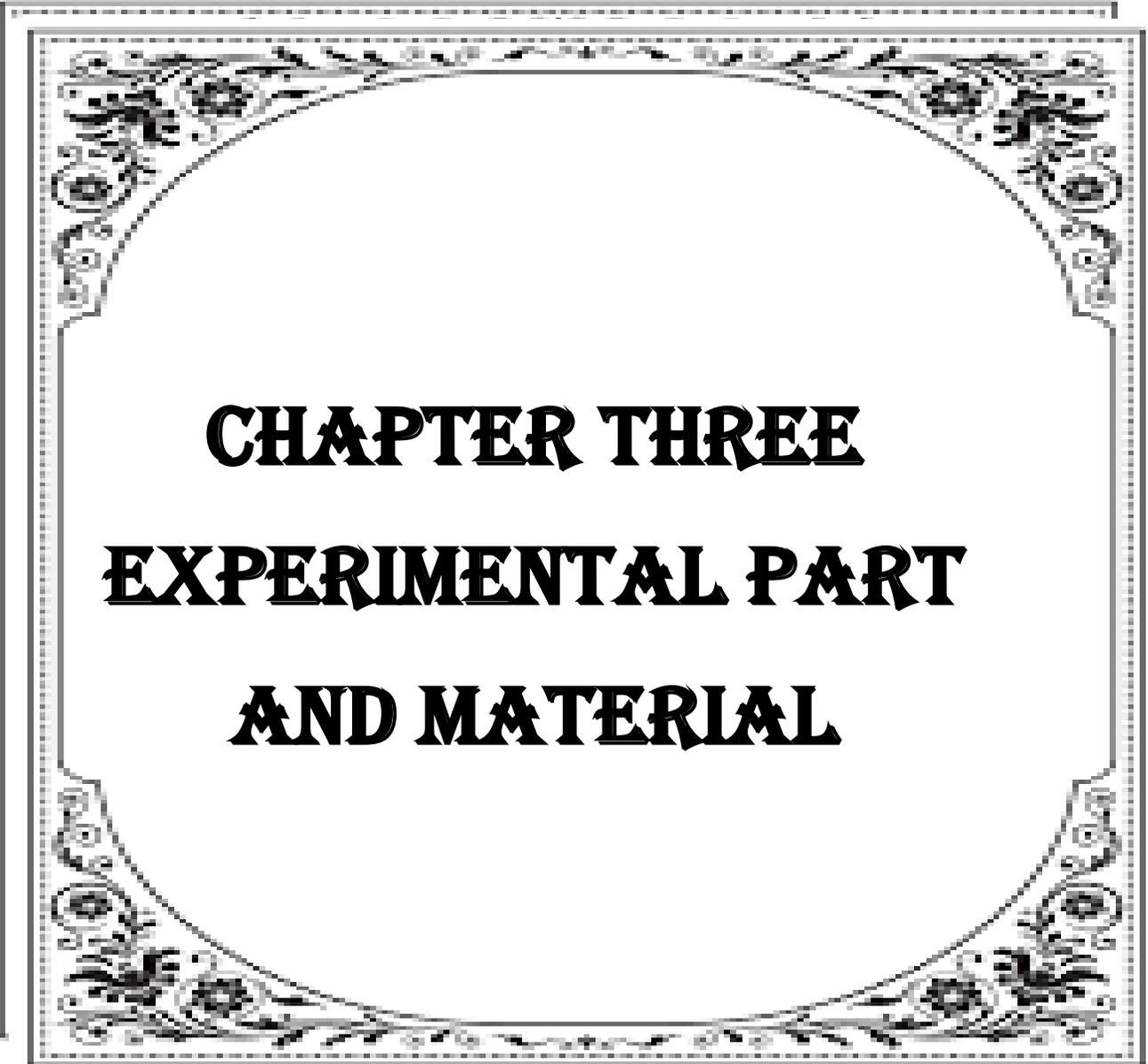


Figure (2.2): The Electronic Transition Types

- (a) Allowed direct transition. (c) Allowed indirect transition.
- (b) Forbidden direct transition. (d) Forbidden indirect transition[74]



CHAPTER THREE
EXPERIMENTAL PART
AND MATERIAL

Chapter Three

3-1 Introduction

This chapter includes steps for film preparation and examinations for optical microscopy , (UV-Visible-Spectrophotometer) , Fourier Transform Infrared Spectrometer (FTIR), Field Emission Scanning Electron Microscopy (FE-SEM) are described. And the materials used in them, in addition to a description of the devices used, Figure (3-1) shows the work stages.

3- 2 Materials used in study:

3-2-1 Ferrite material ($\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$) prepared in college of education for pure science by a master student at particles size(200nm)

3-2-2 Poly Methyl Methacrylate (PMMA) :

The synthetic polymer called Poly Methyl Methacrylate (PMMA) is a colorless transparent thermoplastic that is a white powder with chemical symbol $(\text{C}_5\text{O}_2\text{H}_8)_n$ technically classified as a type of amorphous glass [14] Soluble in organic solvents such as chloroform alcohol has a molecular weight (100.12 g/mol) with a purity of 99.950%. Manufacturer Avonsheim (UK) is preferred because of its mild characteristics, ease of handling and low cost, but it crumbles under load and is more susceptible to scratching than glass the chemical structure of the polymer (PMMA) as shown in Figure (3-1) Their resistance to the surrounding environmental conditions and depending [6]

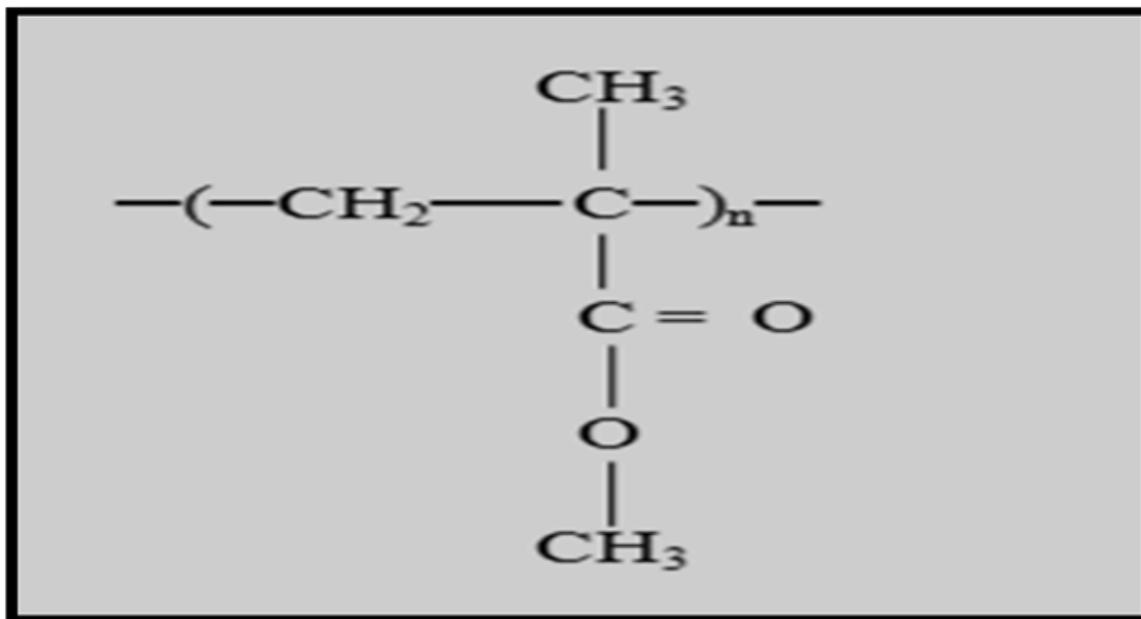


Figure (3-1): Chemical Composition of Polymer (PMMA)[71]

Table (3 - 1): Physical properties of PMMA [4]

Parameters	PMMA
Chemical formula	(C ₅ O ₂ H ₈) _n
Tg	379K , 10 ⁶ °C
Refractive index	1.49
Density (g/cm ³)	1.18
Melting point (K ⁰)	433C ⁰
Molecular weight Mw (g/mol)	100.12 gm/mol

3-2-3 Chloroform or trichloromethane is an organic compound with formula CHCl_3 . It is a colorless, strong-smelling, dense liquid that is produced on a large scale as a precursor to PTFE. It is also a precursor to various refrigerants[72] It is one of the four chloromethanes and a trihalomethane. It is a powerful anesthetic, euphoriant, anxiolytic and sedative when inhaled or ingested[73]

3-3 Tools and Equipment in Preparing Films:

3-3-1 Sensitive electronic balance:

A device used to measure the masses of materials used in the preparation of films equipped with a digital counter with high accuracy to reduce measurement errors. Its trademark is Sartorius.

3-3-2 Magnetic Stirrer:

It is a device used for mixing in chemical solutions and consists of a rotating magnet immersed in the solution and equipped with heat plates to heat the solution when needed. All sample films were prepared at room temperature of 27°C .

3-4 Preparation of Films :

In this study, prepared $\text{PMMA}(\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3)$ $x=2$ nanocomposite films solution casting technique .the First used (1 g) of (PMMA) put into a (500 ml) glass beaker containing (100 ml) chloroform alcohol (CHCl_3) as a solvent, using a magnetic stirrer, at room temperature until complete dissolution. As a result, the magnetic stirrer was continued for 30 minutes until the two substances were completely mixed in the beaker. The solution is poured into a petri dish with a diameter of (9 cm) (clean from dust, dirt and dry) and placed on a horizontal surface and left to dry for one week in the laboratory preservative to keep it from dust and using lukewarm water, the film was obtained from Peter dish(PMMA)

blend was obtained. The same previous steps were repeated with the addition of particles ($\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$) Nano Ferrite with different weight ratios (2,4,6,8%) of the weight used for the first sample. We obtained three films of (PMMA) with ($\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$) Nano Ferrite . The thickness of the dried samples were measured using micrometer . as it is clear in Figure (3-1)

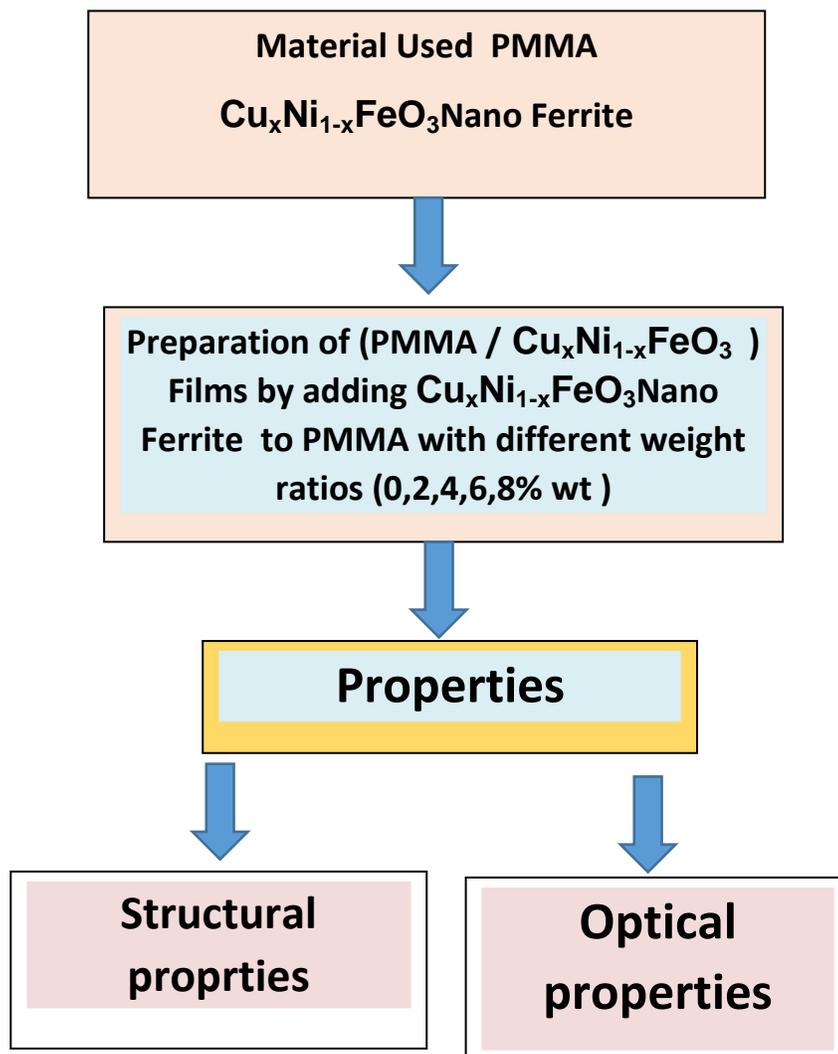


Figure (3-2): The Preparation Condition of (PMMA- $\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$ Nano Ferrite) Films and the Structural, Optical and magnetic field.

3-5 Measurements of Structural Properties

3-5-1 Optical Microscope:

It was used to find out the homogeneity of the prepared films (PMMA) with (FeO₃) nanomaterial and the distribution of the doped within the main compound in this paper Under magnification (4x) as shown in Figure(3- 3), it is located in the Film Lab at the College of Education for Pure Sciences, University of Babylon.



Figure (3-3): Image of optical microscope.

3 -5 -2 Fourier-transform infrared spectroscopy:

FTIR spectra can be used. The quantitative analysis and identification of the recorded nanomaterials (vertex-70 by Brooker of Germany) were in the

wavelength count range (500-4000) cm^{-1} . As shown in Figure (3 - 4), this device was found at the University of Babylon, College of Education for Pure Sciences.



Figure (3-4): Image of FTIR spectroscopy

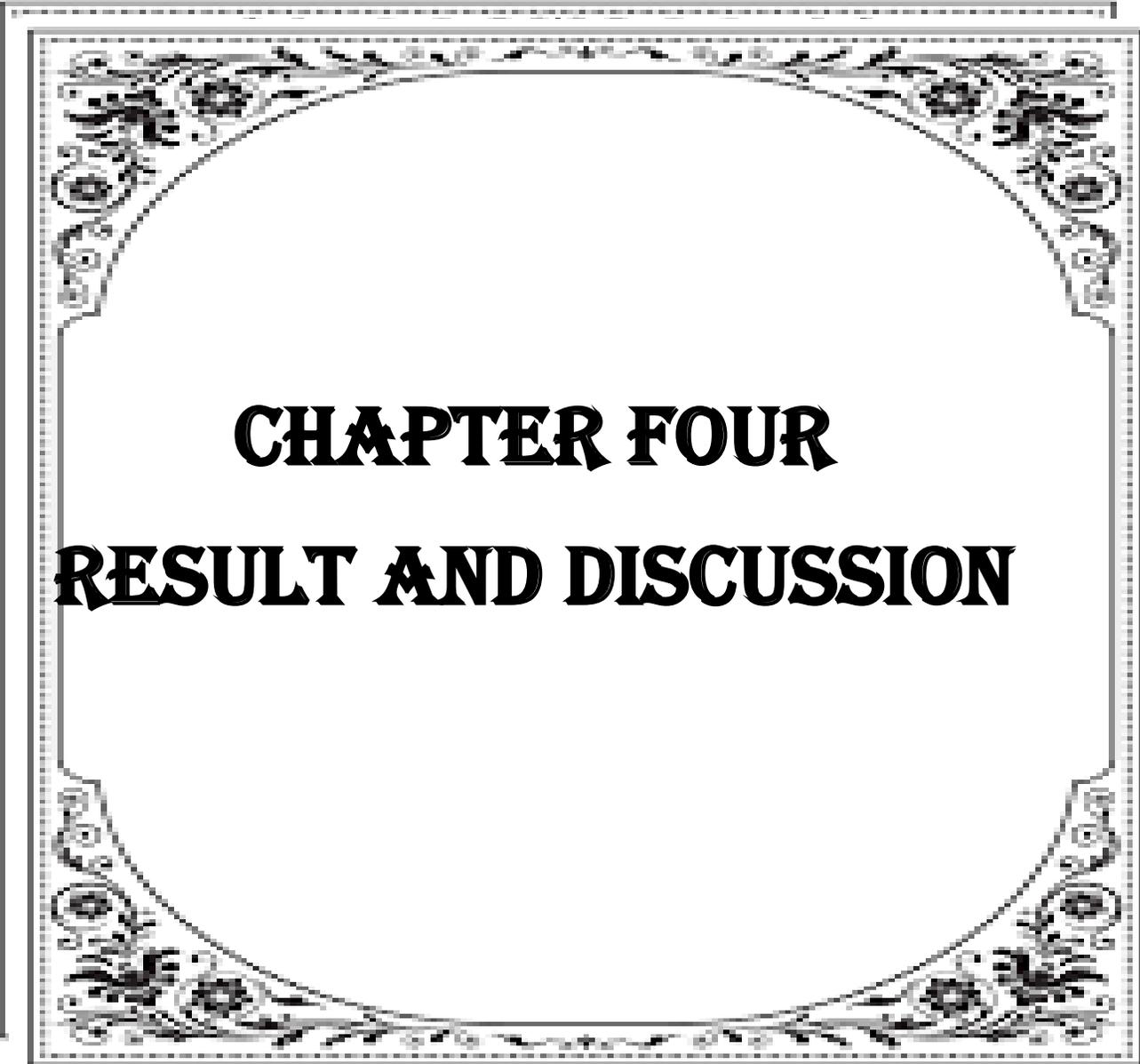
3-5-3 Optical Properties Measurements:

By using a dual-beam spectrometer brand(shimadzu, UV-1800 of Japan origin) with range from(190 to 1100) nm as shown in Figure (3- 5), the absorption spectra of the PMMA / $(\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3)$ nanoparticles films were observed at room temperature and through the use of a computer program (UVP robe software) the optical constants real and imaginary dielectric constant were calculated. Absorbency, absorption coefficient, refractive index, transmittance} as well as

calculation of the energy gap of the prepared PMMA / $(\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3)$ nanoparticles films.



Figure (3- 5):UV photograph for a spectrophotometer



CHAPTER FOUR
RESULT AND DISCUSSION

Chapter four

4-1 Introduction

In this chapter, test results are analyzed on prepared films, analysis of compositional tests (optical microscopy , UV-Visible-Spectrophotometer , Fourier Infrared Spectrometer and Field Emission Scanning Electron Microscopy (FE-SEM) and Optical Properties such as: (Absorption, Transmission, Absorption Coefficient, Energy gap , Refractive Index, Extinction Coefficient and Optical Conductivity) and comparison with the results of previous studies and published research.

4 -2 Structural and Morphological Properties

4-2-1- Field Emission Scanning Electron Microscope (FESEM)

FE-SEM of the (PMMA/Cu_xNi_{1-x}FeO₃) when x=0.2 nanocomposite with different concentration of the ferrite are shown in figure.(4.1). In this Figure, the symbol A,B,C,D and F indicate to PMMA, PMMA/(2%wt.) ferrite, PMMA/(4wt%) ferrite, PMMA/(6wt%)ferrite and PMMA/(8wt%) for (0.2) respectively. From this figure, it is observed that the pure PMMA was homogenous and smooth this indicate a good method for prepared films. Also the uniform morphology dispersed of ferrite inside the PMMA with spherically shaped nanoparticles. The uniform dispersion of nanoparticles in the polymer blend is due to the strong interfacial adhesion between the nanoparticles and blend components. From the figure, it was obtained that the average particle size increased with increasing of concentration of ferrite. This result are agreement with previous studied [74,75].

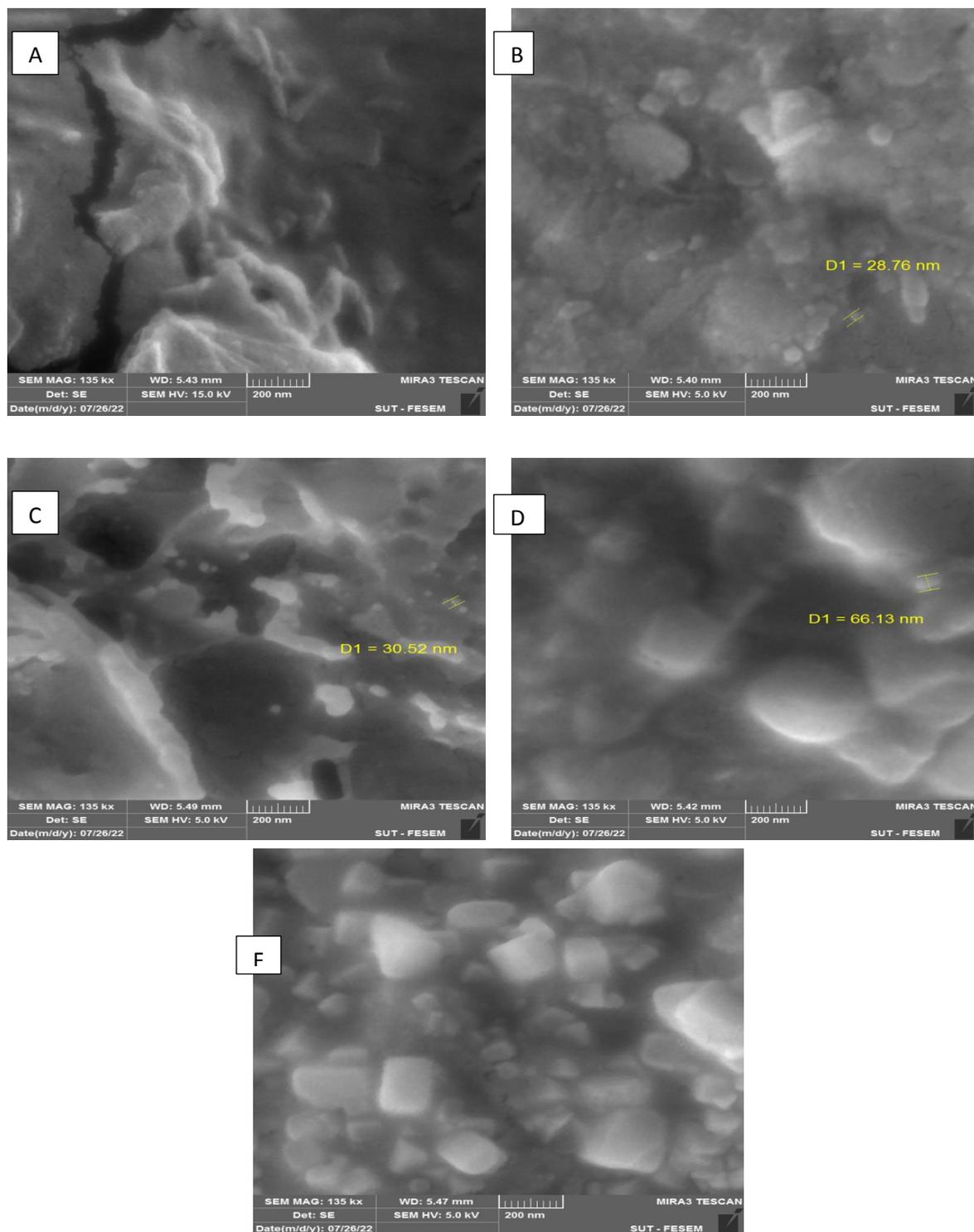
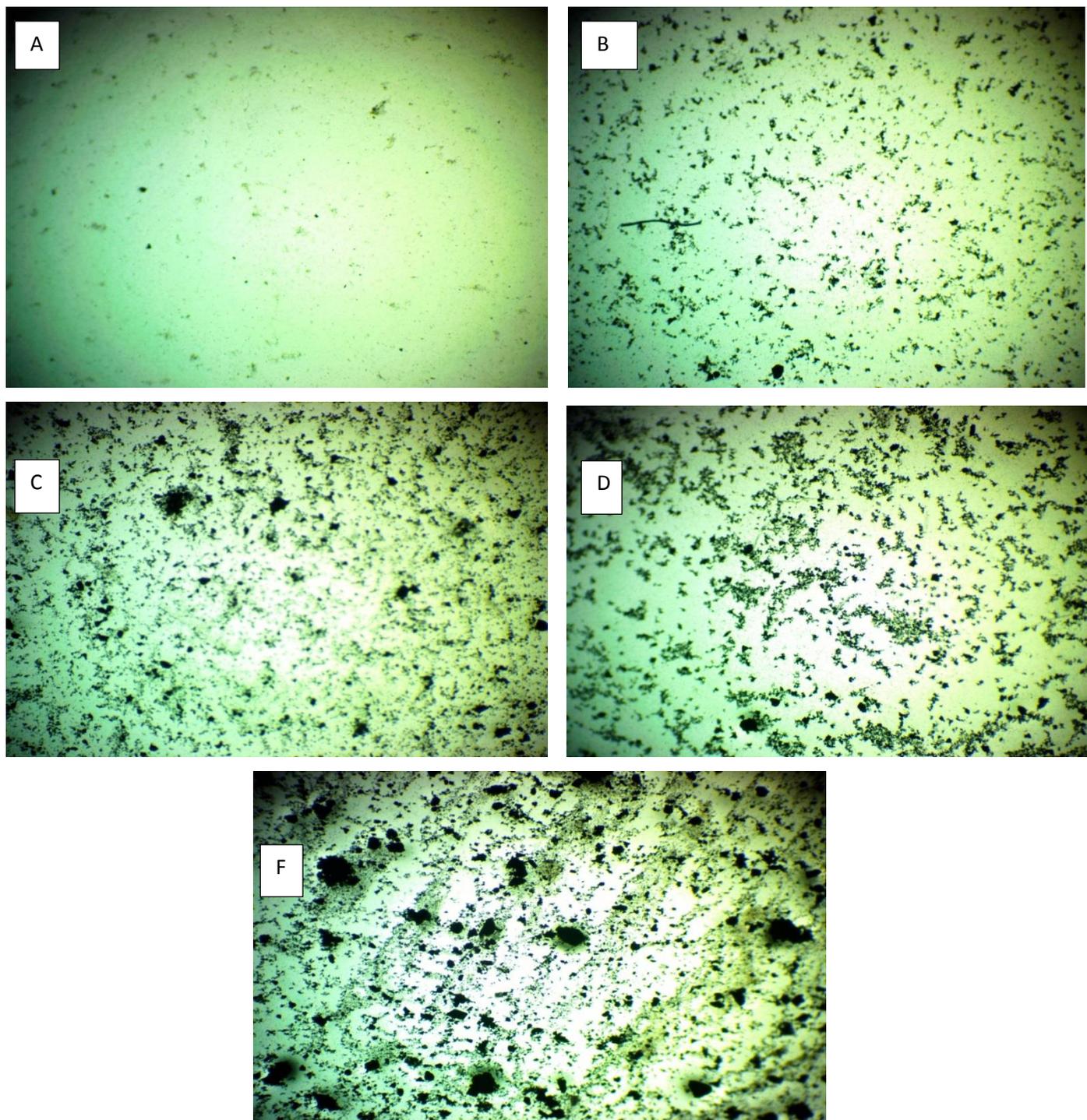


Figure (4.1): FE-SEM images of pure PMMA and PMMA with different ratios(0,2,4,6,8) of Ferrite at $x=0.2$

4 -2 -2 Optical Microscope:

In optical microscopy tests, the surface shape change of the nanocomposites was observed, as all films had a magnification force (4X). Figure (4-2) shows an optical microscope image (A) of (PMMA) mixture before doping and the B, C , D and F images show the surface shape after adding ($\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$) nanoparticles with different weight ratios, and by comparing the pure sample image and the images after adding ($\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$) nanoparticles, It was found that there are many differences between this sample and each of the nanocomposites. Both models demonstrate that when iron trioxide nanoparticles reach a higher concentration, the filler material forms a continuous network within the polymer. Blaine. Paths are formed that allow the charge carriers to pass through them. These results agree with the researcher [76]



Figure(4.2) Photomicrographs (4X) for (A) (PMMA)Pure (B) (PMMA/ FeO_3 2wt.% (C) (PMMA/ Fe O_3 4wt % (D) (PMMA/ FeO_3 6wt%) and (F)PMMA/ Fe O_3 8wt % nanoparticles

4-2-3 Fourier Transform Infrared Analysis:

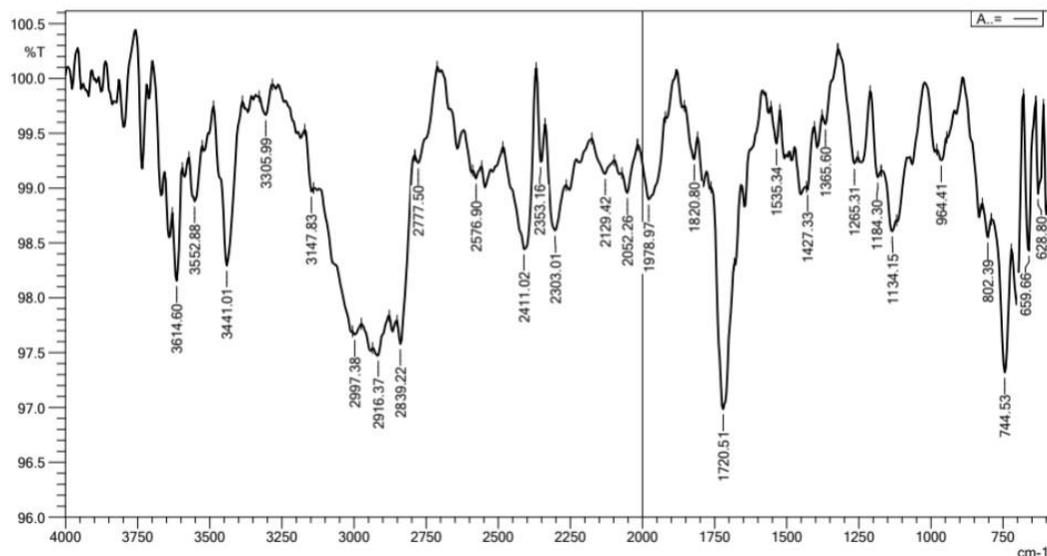
In Figures (4-3A,4-3B, 4-3C, 4-3D,4-3F) show the infrared spectra of PMMA $(\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3)$ nanoparticles films with a range $(500) \text{ cm}^{-1}$. We note the polymer chains correspond to (OH) and (CH) expansion. Fourier transform infrared spectroscopy is often used to analyze materials, to find out the type of polymer, the degree of polymerization, the polymer reactions, and the film components and the vibrational modes of the polymer due to carbonyl and hydroxyl groups as well as hydrogen. In Figure (4-3) shows the Fourier transform infrared spectroscopy of PMMA films, where we note the peaks at $(2949.92, 1723.73, 1449.24, 1240.73, 1145.25, 987.24, 748.34, 696.16) \text{ cm}^{-1}$. The peak of 2949.92 cm^{-1} expresses the stretching band of the hydroxyl group OH, which is a characteristic of alcohols, and the tape represents 1145.25 cm^{-1} for the PMMA crystal and the tape 1723.73 cm^{-1} for the carbonyl group and the tape about 1449.24 cm^{-1} to the bending CH_3 and the range is 987.24 cm^{-1} for the CH_2 vibration[77]

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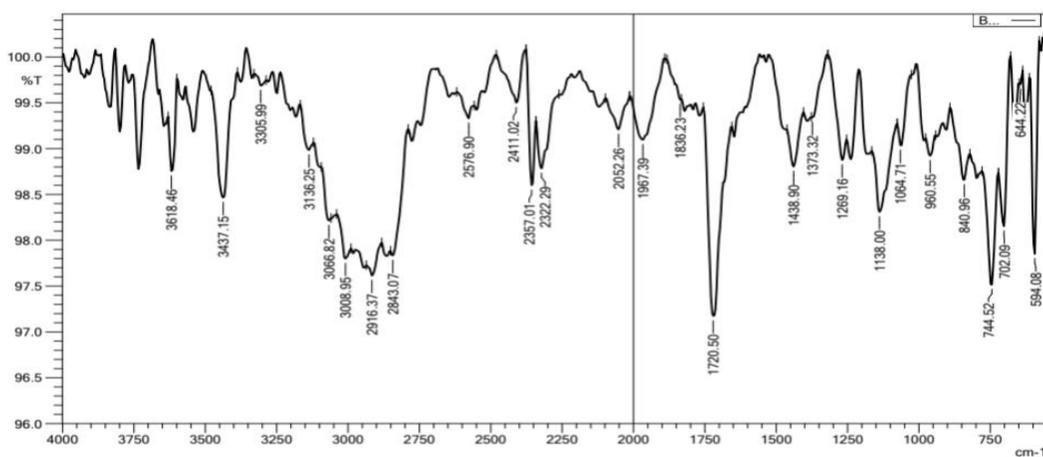
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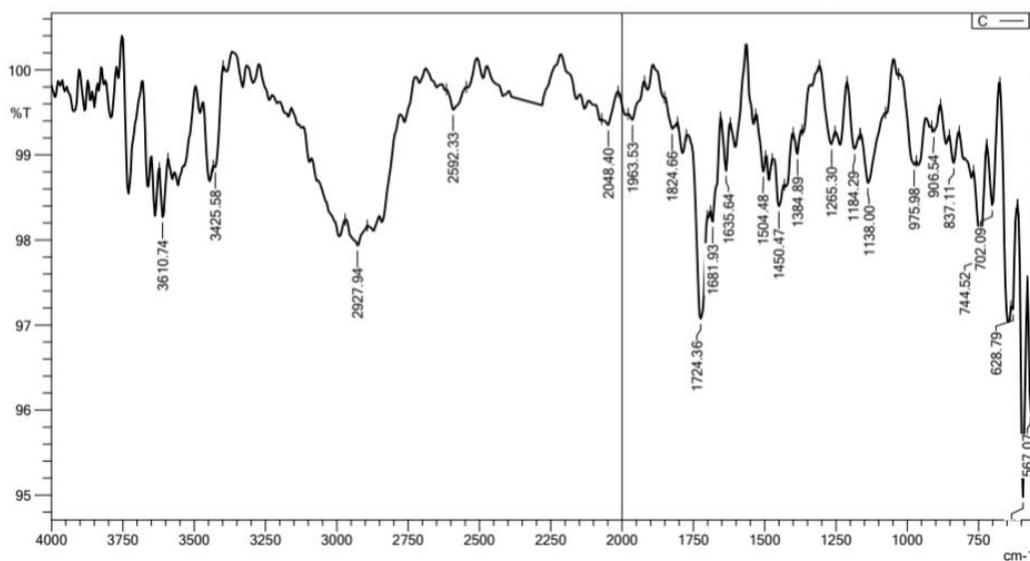
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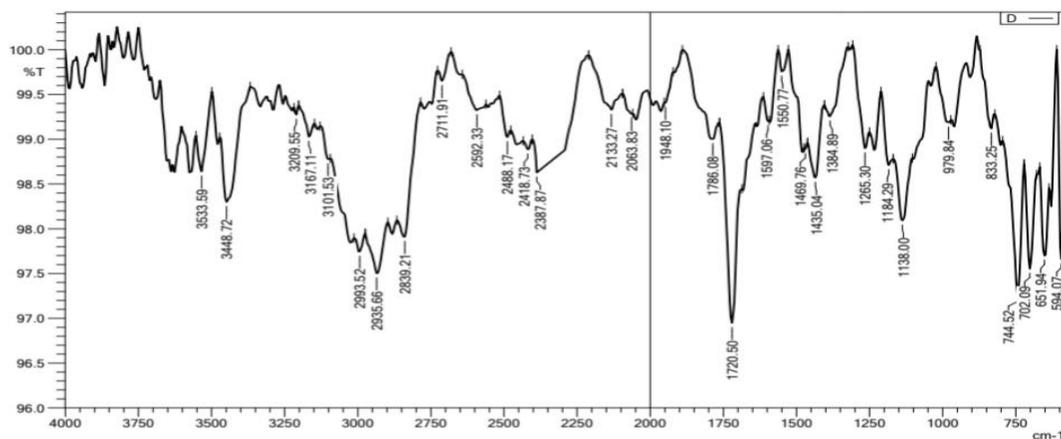
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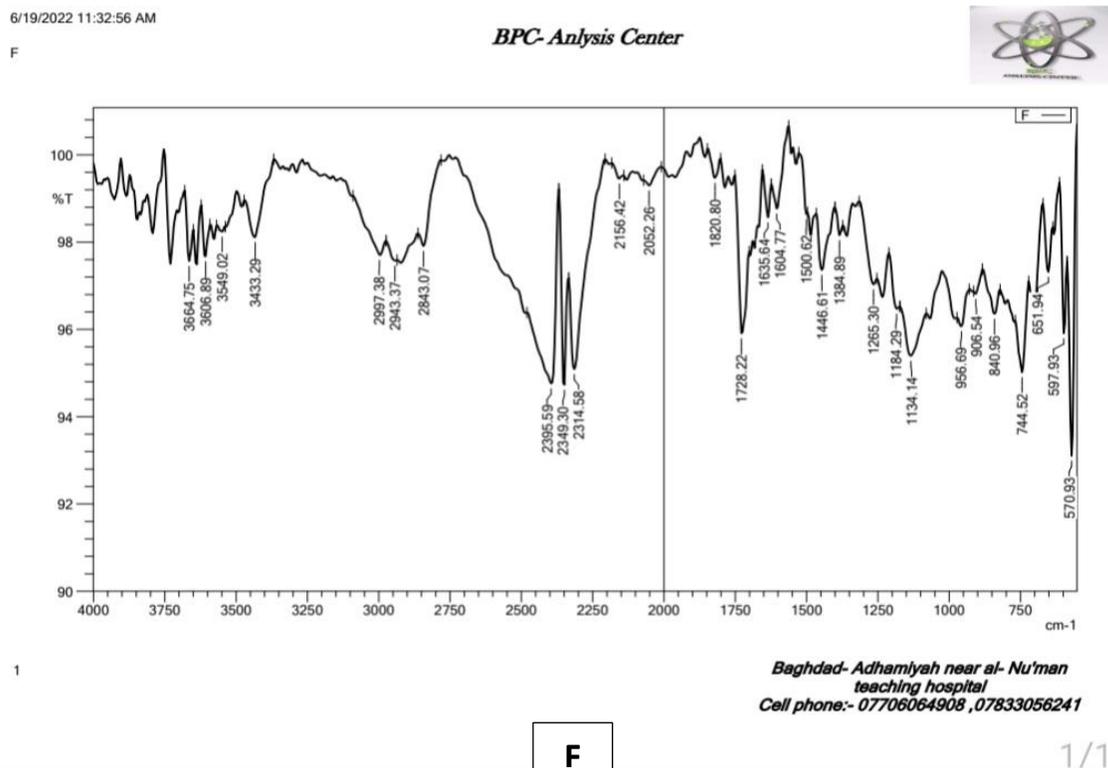


Figure (4-3): FTIR spectra for: (A) PMMA, (B) PMMA –FeO₃ Nanoparticles 2wt.% , (C) PMMA-FeO₃ Nanoparticles 4wt.% (D) PMMA –FeO₃ Nanoparticles 6wt % and (F) PMMA-FeO₃ Nanoparticles 8wt.%

4-3 Optical Properties

4.3.1 The Absorbance of (PMMA/ Cu_x Ni_{1-x} FeO₃) Nanocomposites.

The absorption (PMMA/Cu_x Ni_{1-x} FeO₃) at x=0.2 nanocomposites with different concentrations of ferrite has been recorded at wavelengths range (200-

1100) nm at room temperature. Fig.(4.4) show the absorbance for (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites with wavelength of the light incident respectively. It is observe that the absorbance increase with increasing concentration of the ferrite, while the absorbance decrease with increasing wavelength for all the sample prepared. This is attributed to the excitations of donor level electrons to the conduction band at these energies. Also due to the energy of photon enough to interact with atoms; the electron excites from a lower to higher energy level by absorbing a photon of known energy [78]. This behavior agree with the results of researchers [79-81].

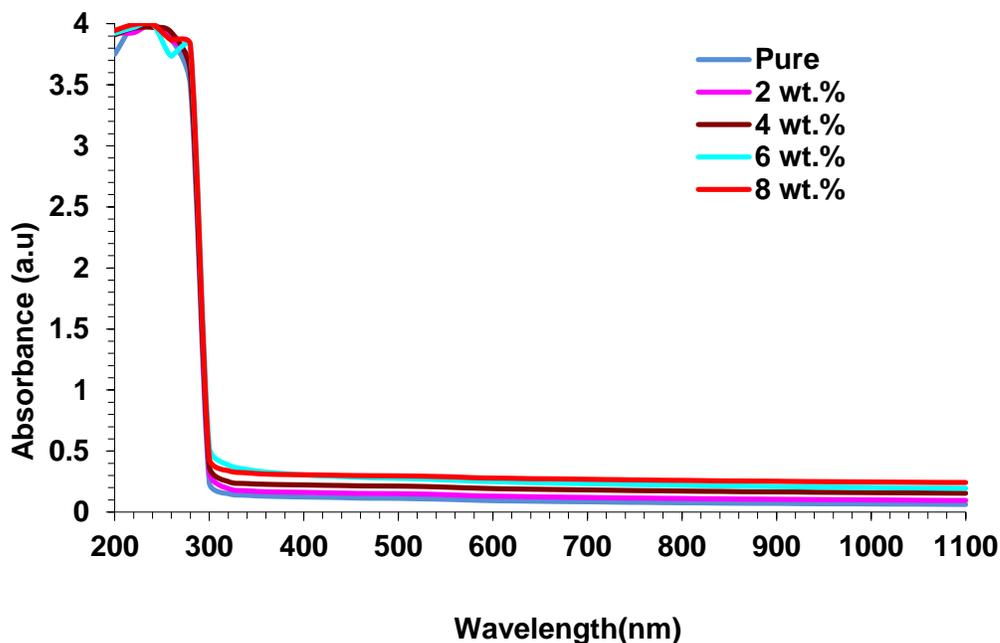


Figure. (4 .4) : The absorbance as a function of wavelength of (PMMA / $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites with different concentrations.

4.3.2 The Transmittance of (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) Nanocomposites.

The transmittance for (PMMA/ $\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$) at $x=0.2$ nanocomposites with wavelength are shown in Fig.(4.5). From this figure shown, the transmittance decreases with the increasing of the concentrations for the ferrite nanoparticles and transmittance increasing with increasing wavelength, This is due to the conglomerate of nanoparticles that occurs when the concentration of nanoparticles increases [82].

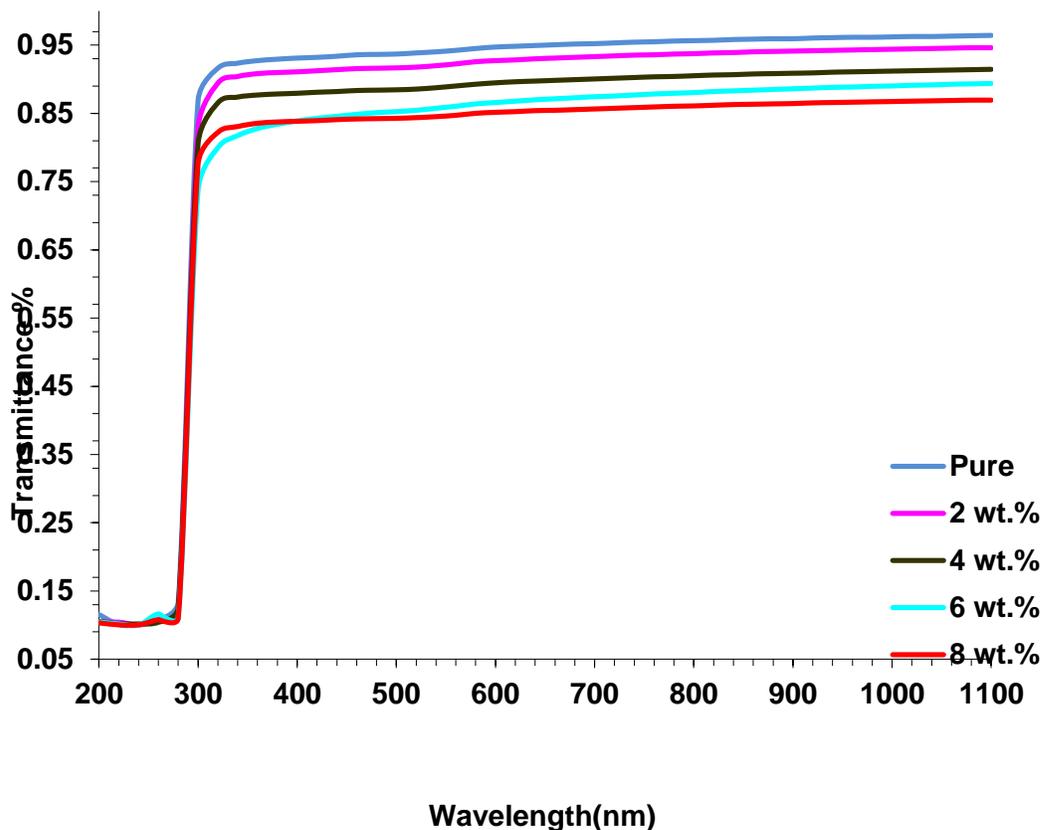


Figure (4.4): The transmittance variation of (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites with the wavelengths.

Optical Parameter:

4.3.3 Absorption Coefficient (α)

The absorption coefficient of Nano composites has been calculated by using equation(2-28)The absorption coefficient versus photon energy of the incident light are shown in Figs.(4.6) for (PMMA/Cu_x Ni_{1-x} FeO₃) at x=0.2 nanocomposites respectively. From this figures, at high energies, the absorption coefficient of all nanocomposites samples is large. This indicates that the electron transition is likely; that is, the energy of the incoming photon is sufficient to move the electron from the valence band to the conduction band, which is possible since the input photon's energy exceeds the energy band gap. The absorption coefficient might help to figure out what kind of electron transition you're dealing with [83,84]. When the material absorption coefficient is large ($>10^4 \text{ cm}^{-1}$), direct electron transition is predicted. When the material absorption coefficient is low (10^4 cm^{-1}), indirect electron transition is predicted. absorption Coefficients of (PMMA/ Cu_x Ni_{1-x} FeO₃) x=0.2 nanocomposites have a low density (10^4 cm^{-1}) and an indirect electron transition. The absorption coefficient of nanocomposites rises as the concentration of ferrite nanoparticles rises, which is due to an augmentation in the number of carriers charge, which raises the absorbance and absorption coefficient for the material (PMMA/ Cu_x Ni_{1-x} FeO₃) at x=0.2 nanocomposites [85].

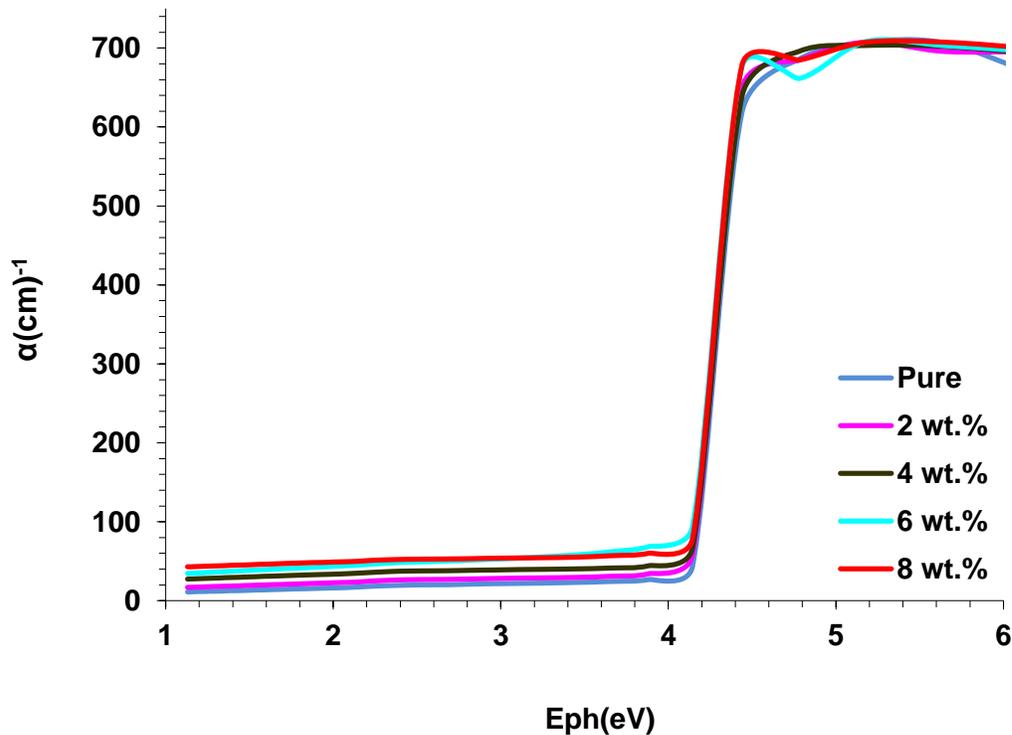


Figure .(4.6) :The absorption coefficient variation of (PMMA/Cu_x Ni_{1-x} FeO₃) at x=0.2 nanocomposite with the photon energies.

4.3.4 Energy gaps (E_g) of the (allowed and forbidden) indirect transition

The energy gap of allowed and forbidden indirect transitions of (PMMA/ Cu_x Ni_{1-x} FeO₃) x=0.2 nanocomposites are shown in figs.(4.7)-(4.8). By drawing a straight line from the top of the curve to the (x-axis) we can find the E_g . The E_g reduce with rise of ferrite nanoparticle content. This attributed to formation of local level in the E_g therefore, the transfer of electron from C.B to local level and then to V.B The value of the E_g of (PMMA/ Cu_x Ni_{1-x} FeO₃) at x=0.2 nanocomposites are listed in Table (4-1). [84]

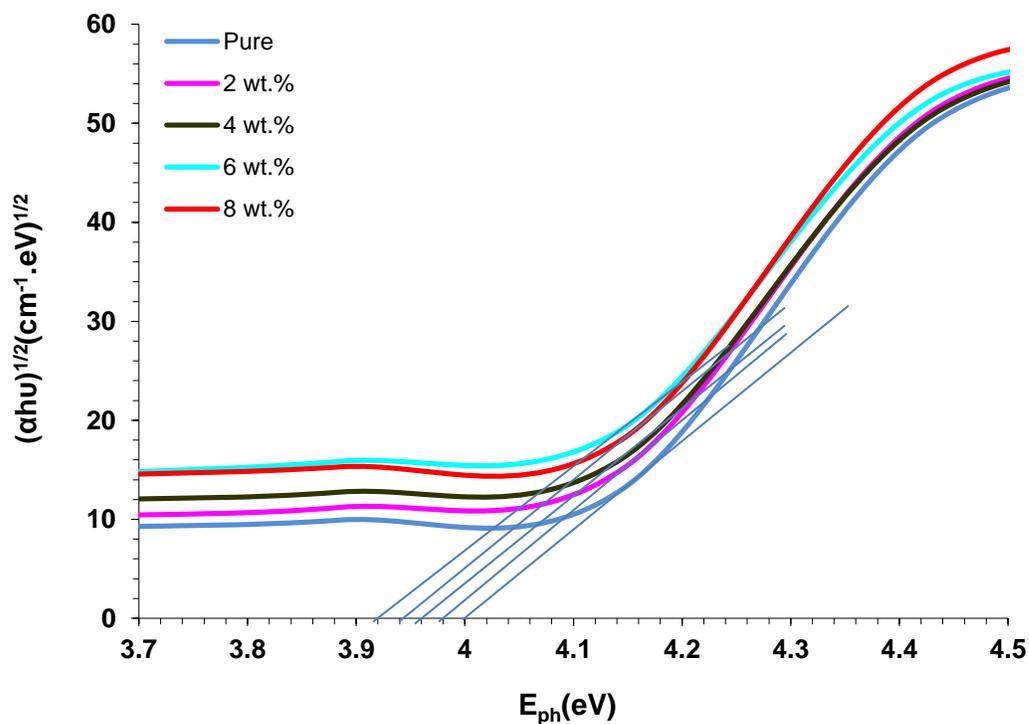
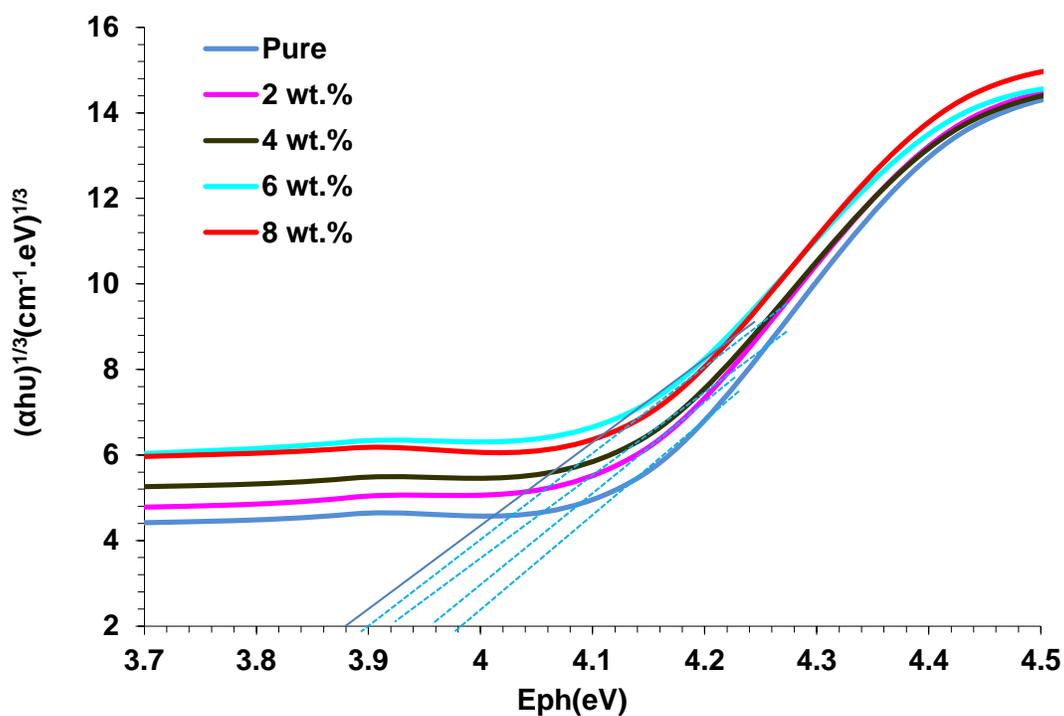


Figure.(4.7): The energy gap for the allowed indirect transition $(\alpha h\nu)^{1/2}$ ($\text{cm}^{-1} \cdot \text{eV}^{1/2}$) versus photon energy of (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposite with different concentration



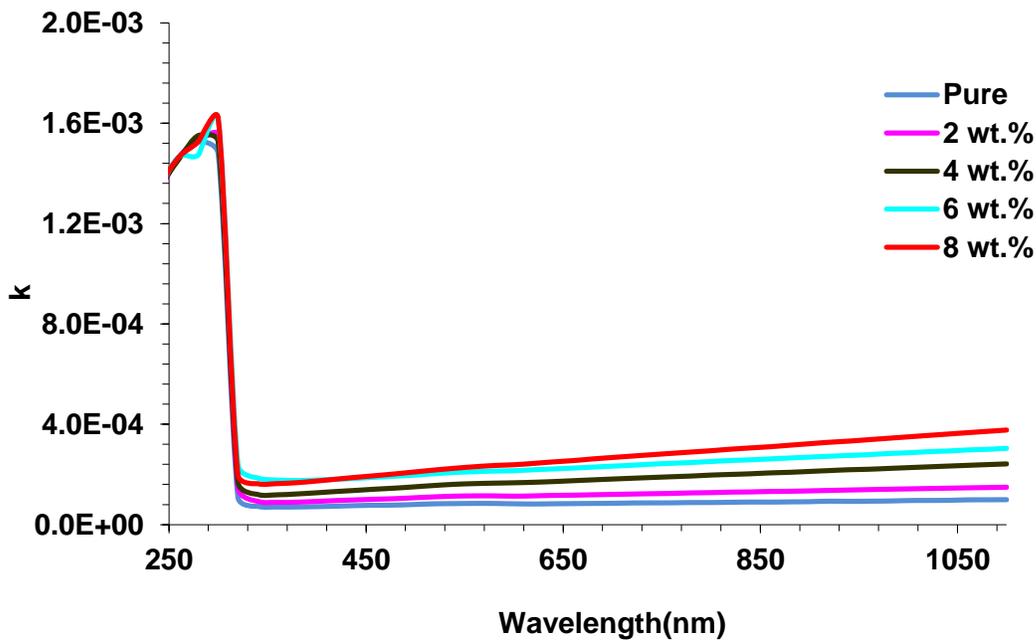
Figure(4.8):The energy gap for the forbidden indirect transition $(\alpha h\nu)^{1/3} (\text{cm}^{-1} \cdot \text{eV})^{1/3}$ versus photon energy of the (PMMA/Cu_x Ni_{1-x} FeO₃) at x=0.2 nanocomposite with different concentration

Table (4.1): The values of optical energy gap for allowed and Forbidden Indirect transitions of (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites.

Ferrite wt% Nanoparticle's concentration	Allowed indirect transition for the (PMMA/(0.2)ferrite)	forbidden indirect transition for the (PMMA/(0.2)ferrite)
0	4	3.98
2	3.98	3.96
4	3.96	3.92
6	3.94	3.90
8	3.92	3.88

4.3.5 The Extinction Coefficient (K) of (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) Nanocomposite

The K were determined from the formula (2- 15). Fig.(4. 9) show the K for (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$)at $x=0.2$ nanocomposites as a function of wavelength respectively. It is observe that K of nanocomposites increases with the increasing of the ferrite nanoparticles concentrations and increase for increasing wavelength, this is due to the increased in absorption optical and photons dispersions in the (PMMA)[85].



Figure(4.9): Relationship between Extinction coefficient for (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) $x=0.2$ nanocomposites with the wavelengths.

4.3.6 Refractive Index (n)

The refractive index is calculated by using equation (2-16). The refractive index of (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites₀ as a function of wavelength is shown in figure(4.10) respectively. It is obtained that the refractive index of nanocomposites rises with ferrite nanoparticle concentration and decreases with wavelength. This is owing to the rising density of nanocomposites in the environment to day. When ultraviolet (UV) light interacts with a material that has a high refractive index, the refractive index of the substance rises [7,87]

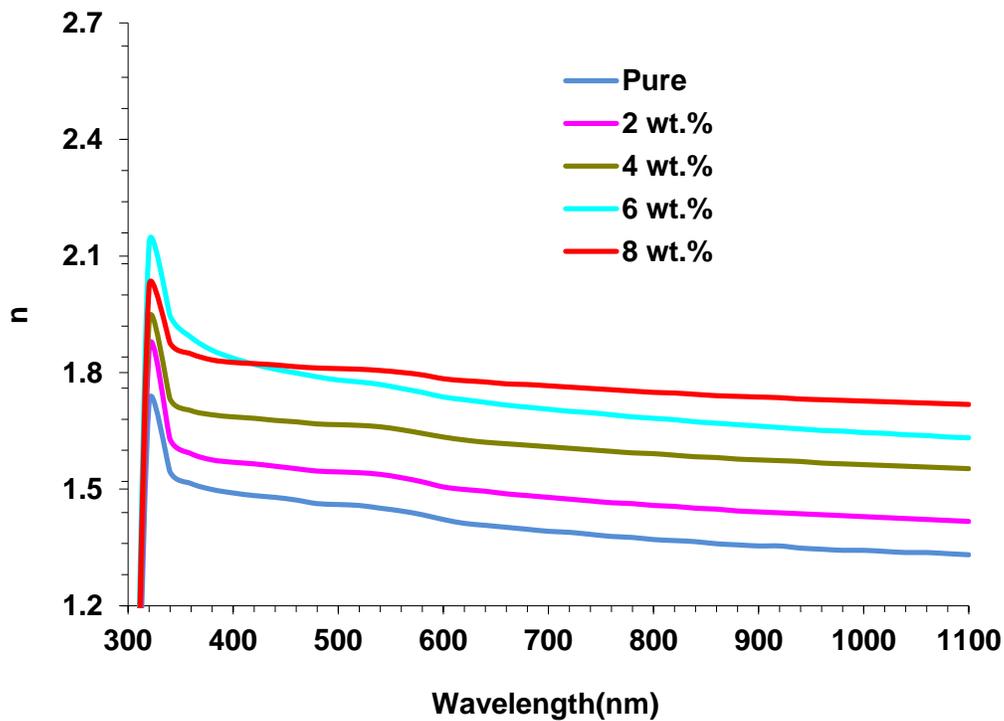
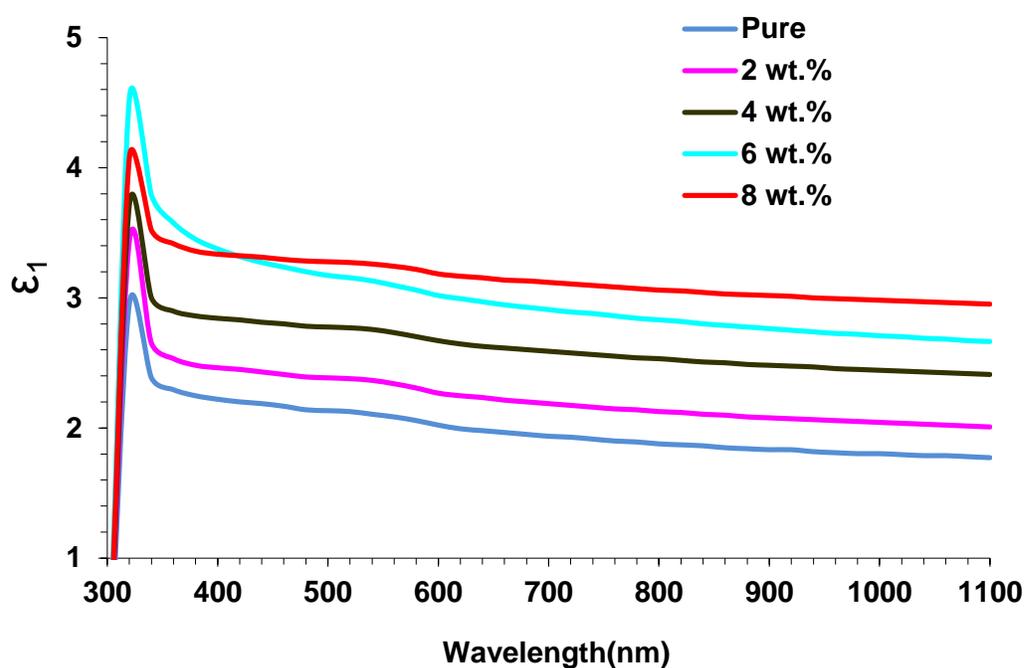


Figure (4.10) : variation of refractive index for (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites with wavelength

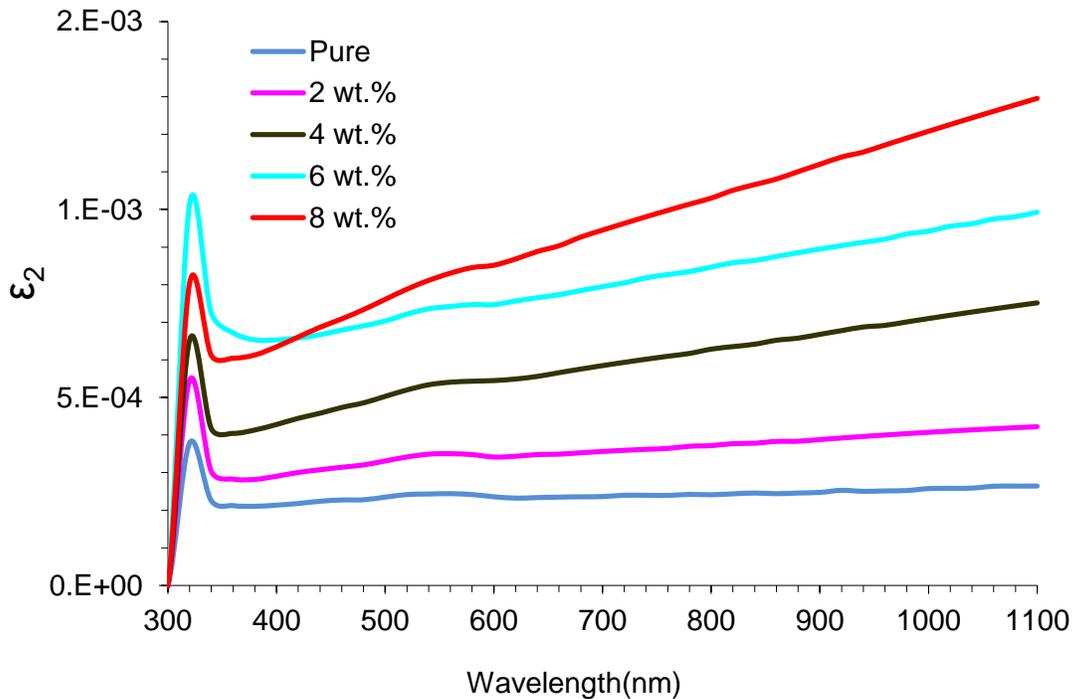
4.3.7 The Real and Imaginary Parts of Dielectric Constant (ϵ_1 , ϵ_2) of (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) Nanocomposites .

By using the equations (2-23) and (2-24), the real and imaginary parts of dielectric constant can be calculated. The ϵ_1 and ϵ_2 versus wavelength for the (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites are shown in Figs.(4.11)-(4.12). From this figures, it is obtain the real and imaginary parts of the dielectric constant of the nanocomposite increase with increasing ferrite nanoparticle concentrations and decrease with increasing wavelength, with the increase in electrical polarization attributed to, the sharing of nanoparticle concentricity in the sample causing this increase, in ϵ_1 , ϵ_2 of the nanocomposite [88]. The ϵ_1 , ϵ_2 of

nanocomposite with change wavelength, as shown in the figures. This is attributed to the refractive index (n) is small, the ϵ_1 depend on n and when extinction coefficient (K) is small, it depends on the ϵ_2 . This is especially true in the near infrared and visible wavelength regions of, where the approximately is n equal while constant the effect of K is very small [89].



Figure(4.11): Real part variation of the dielectric constant of (PMMA / $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites with the wavelength.

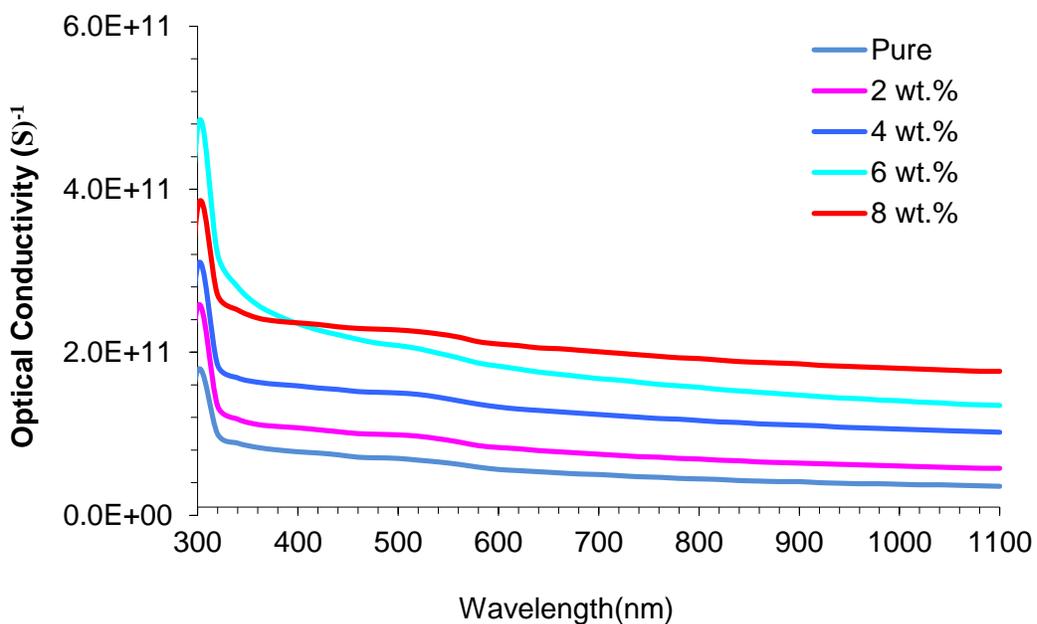


Figure(4.12): Imaginary part variation of the dielectric constant of (PMMA / $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites with the wavelength.

4.3.8 Optical Conductivity (σ_{op}) of (PMMA/ $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) Nanocomposites .

The optical conductivity of the ((PMMA / $\text{Cu}_x \text{Ni}_{1-x} \text{FeO}_3$) at $x=0.2$ nanocomposites with a wavelength are shown in Fig.(4.13) respectively. It is note that the optical conductivity for all sample decreased with the increased in wavelength, this result due to that the σ_{op} depend on the wavelength of the radiations incidental on the nanocomposite specimen. Because the high absorption at lo w photon wavelength, the optical conductivity is increase at this area, the σ_{op} give that all specimens are transmission within visible and near IR range. The σ_{op} for nanocomposites is also increased with an increased concentrations of nanocomposites associated with the establishment of local level concentrations in the energy band, the rise in concentrations of nanocomposites (lead oxide) induced

an increase in the density of localized phases an the band structure; thus, an increase in the absorption coefficient suggests an increase in (σ_{op})of the nanocomposites. This result is agree with researches [90].



Figure(4.13): Optical conductivity variation for (PMMA /Cu_x Ni_{1-x} FeO₃) at x=0.2 nanocomposites with the wavelengths

4.4 Conclusions

The following points are concluded:

- 1- FESEM and optical microscope proved that the ferrite $\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$ with $x=0.2$ forms a continuous network within the PMMA composite
- 2- FTIR obtained that no interaction between ferrite and PMMA polymer matrix and exhibited shifts in certain bonds and changes in intensities.
- 3- The absorbance increases with increasing concentration of ferrite within PMMA polymer matrix, while the transmittance and energy gap decreases with increasing concentration ferrite in the polymer matrix.
- 4- absorption coefficient, extinction coefficient, refractive index real and imaginary dielectric constant and optical conductivity increase with increasing concentration ferrite in the polymer matrix.

4.5 Future Works

The following suggests for future work:

- 1- Studying the effect of ferrite materials on the physical properties for (PVA-PAAm) nanocomposite
2. Effect of the prepared ferrite nanoparticle material on Structural, optical and electrical properties of (PVA)
3. Preparation and studying the thickness effect on attenuation properties for (CMC-PVP-ferrite) within the frequency range (3-8 GHz).

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الخلاصة

حضرت أغشية (PMMA) من متراكبات البوليمر المطاوع للحرارة المسمى (بولي مثيل ميثا اكريليت) مع جسيمات المادة الفيرايتية ($\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$) عند $x=2$ وبنسب وزنية كالتالي (0 , 2 , 4 , 6 , 8)% من الوزن بطريقة صب المحلول وباستخدام كحول الكلوروفورم كمذيب وفي درجة حرارة المختبر لمدة خمسة عشر دقيقة.

استخدم المجهر الضوئي (Optical Microscopic) لمعرفة توزيع المادة الفيرايتية ($\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$) داخل الاغشية المحضرة بصورة متجانسة وعدم وجود تكتلات .

استخدم مطياف تحويل فورييه للأشعة تحت الحمراء (FT-IR) لفحص الاغشية المحضرة حيث لم يلاحظ وجود تفاعل كيميائي بين مكونات المواد التي تم تحضير الاغشية منها .

استخدم المجهر الالكتروني الماسح (FE-SEM) يستخدم لتصوير تفاصيل طبوغرافية صغيرة جدًا استخدم لقياس grain size للمادة الفيرايتية ($\text{Cu}_x\text{Ni}_{1-x}\text{FeO}_3$).

تمت دراسة الخواص الفيزيائية البصرية والتركيبية للأغشية المحضرة باستخدام مقياس طيف الامتصاص ضمن المدى (190 – 1100 nm) والمجهر الضوئي ومطياف فورييه للأشعة تحت الحمراء أثبتت الدراسة ان الخواص البصرية (الامتصاصية ، معامل الامتصاص ، النفاذية ، معامل الخمود ، معامل الانكسار ، فجوة الطاقة) لهذه الاغشية ازدادت عند زيادة النسب الوزنية لجسيمات المادة الفيرايتية ($\text{Cu}_x\text{Ni}_{1-x}$) FeO_3 النانوية بينما تناقصت النفاذية وفجوة الطاقة



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قسم الفيزياء

تحضير واستقصاء الخواص التركيبية والبصرية للمترابك النانوي (بولي مثيل ميثا
اكريليت_ مادة فراييتية)

بحث مقدم
الى مجلس كلية التربية للعلوم الصرفة في جامعة بابل وهي
جزء من متطلبات نيل درجة الدبلوم العالي تربية / فيزياء المواد وتطبيقاتها

من قبل الطالب

حمزة أسماعيل إبراهيم رميض

بكالوريوس علوم فيزياء
الجامعة المستنصرية 2019 م

بإشراف

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