Republic of Iraq
Ministry of Higher Education and Scientific Research
University of Babylon / College of Science
Department of Physics

Green Synthesis of a Composite Based on Polyaniline and Okra Plant Waste for Bio-Medical Applications

A Thesis
Submitted to the Council of the College of Science, University of Babylon in Partial Fulfillment of the Requirements of the Degree of Doctor of Philosophy in Science /Physics

By
Nawras Hofzi Shlioh Shaker

B.Sc. in physics / 2006

M.Sc. in physics / 2019

Supervised by

Prof.Dr.Nihad Abdulameer Salih Kdhair

2022 A.D.   1443 A.H .

بِسْمِ ٱللَّهِ ٱلرَّحْمَٰنِ ٱلرَّحِيمِ

إِنَّا فَتَحْنَا لَكَ فَتْحًا مُّبِينًا ۞ لِّيَغْفِرَ لَكَ ٱللَّهُ مَا تَقَدَّمَ مِن ذَنۢبِكَ وَمَا تَأَخَّرَ وَيُتِمَّ نِعْمَتَهُۥ عَلَيْكَ وَيَهْدِيَكَ صِرَٰطًا مُّسْتَقِيمًا ۞ وَيَنصُرَكَ ٱللَّهُ نَصْرًا عَزِيزًا ۞

صدق الله العلي العظيم

سورة الفتح آية (١-٣)

ACKNOWLEDGMENTS

In the name of Allah (the Almighty) Great is the most gracious and the most merciful. I praise Allah (the Almighty), the lord of earth and heaven for completing my thesis.

Many thanks to the Deanery of the College of Science in University of Babylon and the Department of Physics for offering me the opportunity to complete my study.

I would like to extend my thanks and gratitude to my supervisor, ***Prof. Dr. Nihad Abdulameer Salih*** for having suggested this topic for me and for his extinguished efforts, sound advice and directions that have helped in smoothing out every difficulty encountered during my work.

I deem it an honour to offer my thanks and gratitude to ***Prof.Dr. Mohammed Almaamori*** and ***Assistant.Prof.Dr. Hikmat Adnan Jawad***, for the valuable suggestions. Special thanks to ***Assistant Prof. Dr. Saba Abdul Zahra Obaid*** and ***Assistant.Prof. Dr. Ahmed Kadem Kodaery*** of their cooperation and help during the course of My study.

Last but not least Iam very grateful to my family for their consistent encouragement support during my study, them have guided and changed my life with brighter and prosperous future. Without them, I will not be what Iam today. I love them all.

Nawras

DEDICATION

I dedicate this effort to

Dears

My mother,

My sisters, my brother,

My husband,

My daughter and my sons

with all my gratitude

Nawras

Summary

In this work, synthesis of a composite based on polyaniline and okra plant waste are prepared using spin coating devise. The samples prepared from Polyaniline (PANI), okra plant waste and silver (Ag) nanoparticles. Thin films of (PANI/okra plant waste) blend, okra plant, PANI and (PANI/okra plant waste/Ag) nanocomposites were prepared with Ag nanoparticles added to the (PANI/okra plant waste) blend at different weights (0.5%, 1%, 1.5% and 2%). The effect of okra plants on the structural, D.C electrical and optical properties of (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites were studied for antibacterial activity, electrodes , polymer eyeglasses lens and gradient-refractive index lens (GRIN) lenses applications.

The optical properties of (PANI/okra plant) blend and (PANI/okra plant/Ag) nanocomposites were studied in the wavelength range (300-1100) nm. The results indicated that the optical parameters of (PANI/okra plant) blend were enhanced with the added of the okra plants. The results showed that the absorbance increases in the (PANI/okra plant) blend. The absorption coefficient and optical energy gap were also calculated, and the results showed that the polymer possesses indirect permissible energy gap and that it decreases by added the okra plants to the formation of localized levels in the energy gap. The D.C electrical properties of (PANI/okra plant) blend and nanocomposites where the results showed that the electrical conductivity increases with increasing temperature and also by added the okra plants.

The results of application of (PANI/okra plant) blend films as electrodes material showed that the electrical resistance decreases with plants waste additives to the polymer. These findings provide insights for the development of new electrodes to recording of electrocardiography (ECG), electromiography (EMG) and electroencephalography (EEG) signals.

The results of polymer eyeglasses lens and gradient-refractive index lens (GRIN) lenses applications for (PANI/okra plant) blend and nanocomposites show that in their optical behaviors have better refractive and improve transparency. Thus, the use of plants waste based on polymeric material results in different properties in the prepared material from plants waste, when used in the polymer lenses applications.

The antibacterial properties of the (PANI/okra plant) blend and (PANI/okra plant/Ag) nanocomposites have been tested against pathogens (Staphylococcus epidermidis as Gram positive bacteria and klebsiella pneumoniae as Gram negative bacteria). Nanocomposite and (PANI/ okra plant) blend presented antibacterial activity against tested bacteria. Films contained okra mucilage showed more antibacterial activity. The inhibitory activities of resultant films were stronger against klebsiella pneumoniae than Staphylococcus epidermidis.

Contents

List of Symbols

Symbol	Physical meaning
A	Absorptance
α	Absorption Coefficient
c	Speed of Light in Vacuum
E_e	Electric Field
E_f	Fermi Energy
E_g	Energy Gap
E_{act}	Activation Energy
E_p	Photon Energy
h	Plank Constant
I_A	Absorbed Light Intensity
I_o	Incident Intensity of Light
I_T	Transmittance Photon Intensity
J	Current Intensity
k_o	Extinction Coefficient
K	Wave Vector
k_B	Boltzmann's Constant
n	Refractive Index
e	Charge of Electron
R	Resistance of the Film
$\varepsilon_i, \varepsilon_r$	Imaginary and Real Part of Dielectric Constant
T	Transmittance
V	Applied Voltage
υ	Frequency
ρ	Resistivity

Symbol	Physical meaning
σ	Electrical Conductivity
σ_o	Minimum Electrical Conductivity
$\sigma_{d.c}$	D.C Electrical Conductivity

List of Abbreviations

Symbol	Physical meaning
AFM	Atomic Force Microscope
Ag	Silver Nanoparticles
C.B.	Conduction Band
ECG	Electrocardiography
EEG	Electroencephalography
EMG	Electromyography
GRIN	Gradient-Refractive Index Lens
MHA	Mueller Hinton agar
PANI	Polyaniline
PEs	Polymer electrolytes
RMS	Root Mean Square
Sa	Mean Height
Sz	Maximum Height
SEM	Scanning Electron Microscope
SC	stratum corneum
VIS	Visible Spectrum
V.B.	Valence Band
UV	Ultra Violet Spectrum

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Chapter One

Introduction and Literature Revi

1.1 Introduction

Polymeric materials have gained a lot of attention in recent years due to their wide range of applications in various fields of life, including medical, engineering, and industrial, due to the fact that the polymer possesses unique properties like optical, mechanical and electrical properties that ensure ease of handling industrial [1]. A polymer is large molecule that contains hundreds or thousands of atoms formed by combining one or two or sometimes more small (monomers) types of the molecule into a chain or network structure [2]. One of the most important means of developing new materials is polymer blending. Blending multiple polymers with a combination of properties improves on the individual polymer components. Generally, polymer materials may by excellent insulators that can by mixed with conductive fillers to improve their conductivity [3].

polymers containing natural fibers have obtained considerable attention. The interest in the natural fiber reinforced polymer arises rapidly due to the high performance physical properties and low cost low density. Natural fibers are renewable, cheaper, pose no health hazards and finally provide a solution to environmental pollution by finding new uses for waste materials. Furthermore, natural fiber reinforced polymer composite form a new class of materials which seem to have good potential in the future. When these fibers are incorporated polymer matrix, they would yield materials with better properties suitable for various applications [4]. Application In industrial and biomedical for their superior characteristics [5].

1.2 Polymer Structure

Polymers are made up of big organic molecules and small structural components (monomers) that are joined together in a polymerization process. Thousands of atoms are joined by covalent chemical bonds in each molecule.

Molecules in a polymer are attracted to one another by forces that vary depending on the polymer type. Polymers are made up of large coupled molecules that are difficult to regulate, structure a linear chain of molecules can arrange themselves in an organized form [6].

1.3 Conductive Polymers

Conductive polymers are a new group of synthetic polymers that combine the chemical and mechanical properties of polymers with the electronic properties of metals and semiconductors [7]. Polymers are usually insulators, but some polymers have been synthesized with a remarkable ability to conduct electricity [8]. These polymers are manufactured to be coming conductive from the reaction of conjugated polymers with an oxidizing agent, reducing agent or an acid. The conductivity of these materials can be adjusted according to the nature of doping, the degree of doping and blending with polymers the other. In addition to the important properties mentioned above, polymeric materials are generally characterized by their light weight, ease of manufacture, and flexibility [9].Conductive polymers are widely used in a wide range of applications. Tissue engineering, regenerative medicine, biosensors and other medical applications are examples of biomedical applications that could be revolutionized by conductive polymers [10].

1.4 Conducting Polymer Synthesis

1- **Chemical Synthesis**: Chemical polymerization is frequently the use of relatively strong chemical oxidants like aluminum peroxide or hydrogen peroxide. These oxidants have the ability to oxidize monomers in solution, resulting in cationic radicals [11].

2- **Electrochemical Synthesis**: is the favored method to obtain a more repeatable arrangement of thin films and polymers [12]. The ability to choose the proper electrolyte material, as well as a considerably larger

selection of cations and anions for use as doping ions, are all advantages of electrochemical polymerization. With polymerization, doping and curing happen at the same time [11].

1.5 Polymer Blend

Almost five decades ago, the field of polymer blend has been a major subject in development and polymer research, it has been the focus of attention for a long time. At the end of the year (1960), the industrial and academic trials in the polymer blend increased significantly [13]. The polymer blend is defined a mixture of at least two different polymers to make a new type of material with different physical properties. The performance of polymer blends depends on the morphology and characteristics of each polymer in the mixture and its content. The cost of the mixture depends on the method of the installation, the morphology of the mixture, and the materials that can be designed for a specific application. Most blends have been developed to improve a specific characteristic such as extending the performance of engineering resins, or the strength of the impact [14].

Nowadays, the market pressure is so high that producers of plastics and other products need to provide the best economic and best materials with excellent combinations of properties as an alternative to traditional metals and polymers [15]. Three types of blends can be classified into; i) fully miscible blends, ii) partially miscible blends, iii) completely immiscible blends. The fully miscible blends consist of a homogeneous single phase. Partially miscible blends are partially mixed, as part of one of the blend components is dissolved in the other, which shows normally good compatibility and fine phase morphologies. However, the completely immiscible blends exhibit the coarse phase morphology with a sharp interface and poor adhesion between the two blend phases. This is the reason for the observed poor characteristics of the

immiscible blend, which depend heavily on the size and distribution of phases[16]. The performance of a polymeric material can be preferable by selecting the appropriate components and their proportions. Polymer blending adds some new properties that lead to the construction of new materials with improved chemical, physical and mechanical properties [17].

1.6 Natural Fiber

In recent years, polymer containing natural fibers have obtained considerable attention. The interest in the natural fiber reinforced polymer arises rapidly due to the high performance in mechanical properties and low cost. Natural fibers are renewable, pose no health hazards and finally provide a solution to environmental pollution by finding new uses for waste materials. Furthermore, natural fiber reinforced polymer composite form a new class of materials which seem to have good potential in future as a substitute for wood based materials in structural applications [4].

The electrical conductivity of these polymers can be improved by using natural fibers, when these fibers are combined in polymer matrix they will produce materials with better properties suitable for different applications [14]. Moreover, the use of natural fibers as reinforcement for polymer-based composites has been attracting the interest of the scientific community for a long time and constitutes a new class of materials that have good potential in the future as many natural fiber polymer composites have found applications as a very important industrial resource in such fields as scaffolds, optical devices, pharmaceutical products, substitutes/medical biomaterials and tissue repair (wound dressing) [4,18]. Plant-based natural fiber, a high potential field of the reinforced polymer composite material, is considered as lightweight and economical products as they possess lower density and extraordinary molding flexibility [19].

Okra is one of the plant fibers that contain various natural materials, the most important of which are cellulose and lignin, which act as a support material for plant fibers [20].Where was use this fiber in composite materials decades for their relatively recyclability, biodegradability and minimal health hazard issues and environment-friendliness compared to the traditional artificial materials (aramid, glass, and carbon fibers) [21,22]. Thus the electrical properties of polymer composites reinforced with natural fibers is suitable for electrical applications as terminals, connectors, industrial sockets, etc. [4].

1.7 Nanomaterials

The nanomaterials are the materials with less than 100 nm size ones at least in one dimension. The nanomaterials show different physicochemical properties than the bulk material which depends on their size and shape. Nanomaterials may be of different shapes like nanorods, nanoparticles, nanosheets which can be characterized based on their dimensionality. By the interaction of two or more particles, their physical properties will alter. These particles of different constituents are called bulk or three-dimensional nanomaterials [23]. The nanomaterials are of different types, they are carbon-based nanomaterials, metal nanoparticles, semiconductor nanomaterials, polymeric nanomaterials and lipid-based nanomaterials [24].

Based on the dimensions of nanoscale (<100 nm) they are classified as follows [23]:

1- Zero-dimensional materials where all dimensions are measured at the nanoscale. Nanoparticles will come into this classification.

2- One-dimensional materials outside the nanoscale, one-dimensional nanomaterials represented by tubes Nanoparticles and nanowires.

3- Two-dimensional nanomaterials represent nano films, nano-layers and nano-coatings.

4- Three-dimensional nanomaterials are not limited to the nanoscale in any dimension. Figure (1.1) showing the classification of nanomaterials based on Dimensions [25].
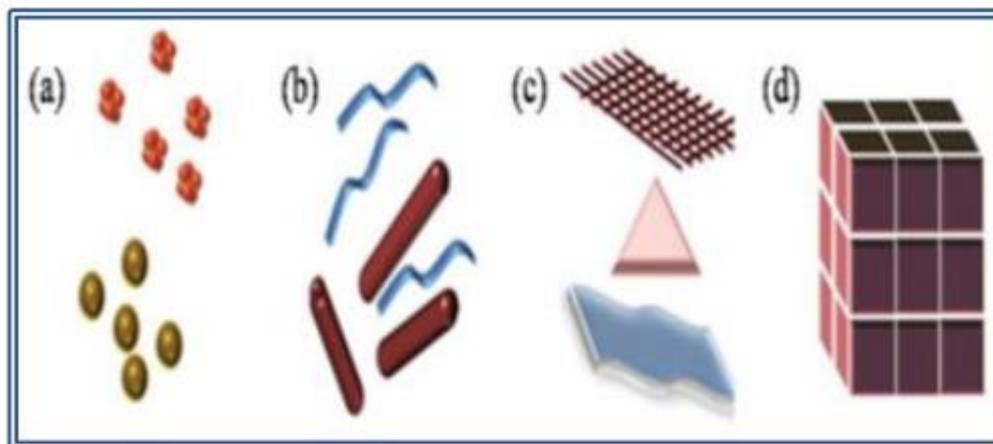
Figure (1.1): Shows the classification of nanomaterials (a) Zero-dimensional nanomaterials (0-D), (b) One dimensional nanomaterial (1-D), (c) Two-dimensional nanomaterials (2-D), (d) Three dimensional nanomaterials (3-D).

1.8 Nanocomposites

Polymer nanocomposites can be expressed as materials contain a tiny quantities of nanoparticles (fillers) distributed in polymers homogenously with different concentrations. In nanocomposites the interaction of fillers with polymers is expected to be much more [26,27]. Nanocomposites are a novel fabrication materials refer to a hopeful field in the area of nanoscience. Besides these properties, they show the amazing advantages of structural, electrical, optical, biocompatibility and biodegradability in various industrial, medical and packaging applications [28].

Inside polymer blend the filler of nanoparticles interaction between them to form molecular bridges for nanocomposites. This is the origin for improved structural, electrical and optical properties of the nanocomposite [29]. Polymer nanocomposites consists of three major substances are the matrix,

reinforcement, and the interfacial region as shown in the Figure (1.2). The last is in charge of incorporation between the matrix and nanofiller [30].
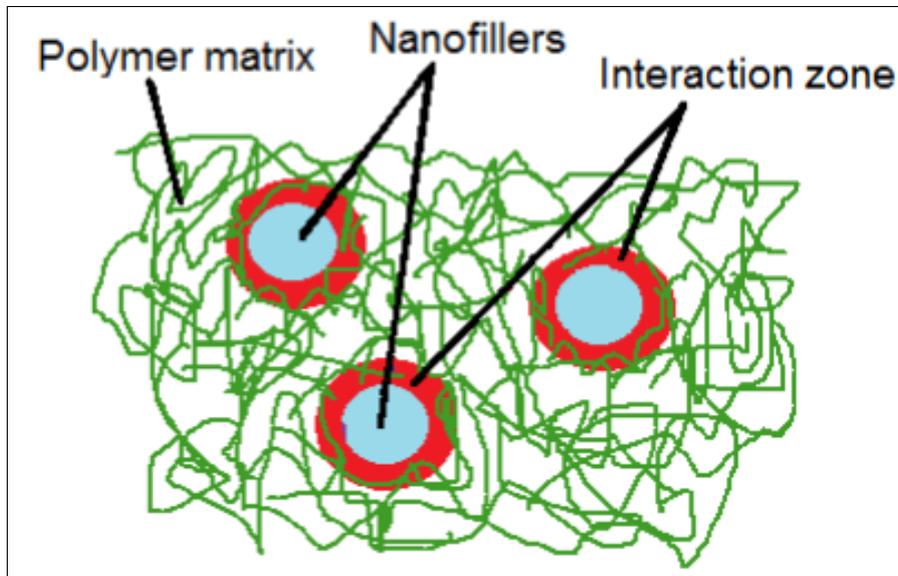
Figure (1.2): The components of polymer nanocomposites [30].

Nanocomposites are classified according to the types of reinforcement materials and matrix materials used in their construction. According to the type of matrix material, nanocomposites are generally classified into following three classes [31]:

1- Polymer matrix nanocomposites.

2- Ceramic matrix nanocomposites.

3- Metal matrix nanocomposites.

Nanocompsites applications are very promising materials in the areas of the microelectronic manufacturing, packing materials, automobiles, integrated optical circuits, drug delivery, injection molded products, coating, fire retardants, sensors and consumer goods….etc [32].

1.9 The Properties of the Used Materials

1.9.1 Polyaniline

Polyaniline (PANI) was discovered by Runge the name of aniline black in 1834 [33]. PANI was identified to be a mixed oxidation-state polymer composed of reduced benzoid units and oxidized quinoid units by Woodhead and Green in 1912. Furthermore, under specific experimental settings, PANI was discovered to have conductor-insulator switching capabilities. Since then, the substance has attracted a lot of attention from researchers [34].

PANI is the most conductive polymer in its chain, giving it distinctive physical and chemical features such as excellent electrical conductivity (p-type) due to the presence of (-NH)-reactive groups. PANI is utilized in a variety of applications, including sensors, electronic devices and batteries. PANI is also inexpensive and light in weight. Because of its remarkable stability, PANI has a unique property in comparison to the remaining conductive polymers in terms of composition, as it comprises nitrogen atoms that represent the distinguishing bridge in the structure of the polymer's backbone. This made it a particularly effective electronic material. The structural formula of the PANI polymer $(C_6H_5NH_2)$, which is shown in figure (1.3) [34]. The oxidation states of polyaniline bases are (LB) (total reduction state), (EB) half-oxidation state, and (PB) total oxidation stat [35].Table (1.1) shows the different types of PANI [36].
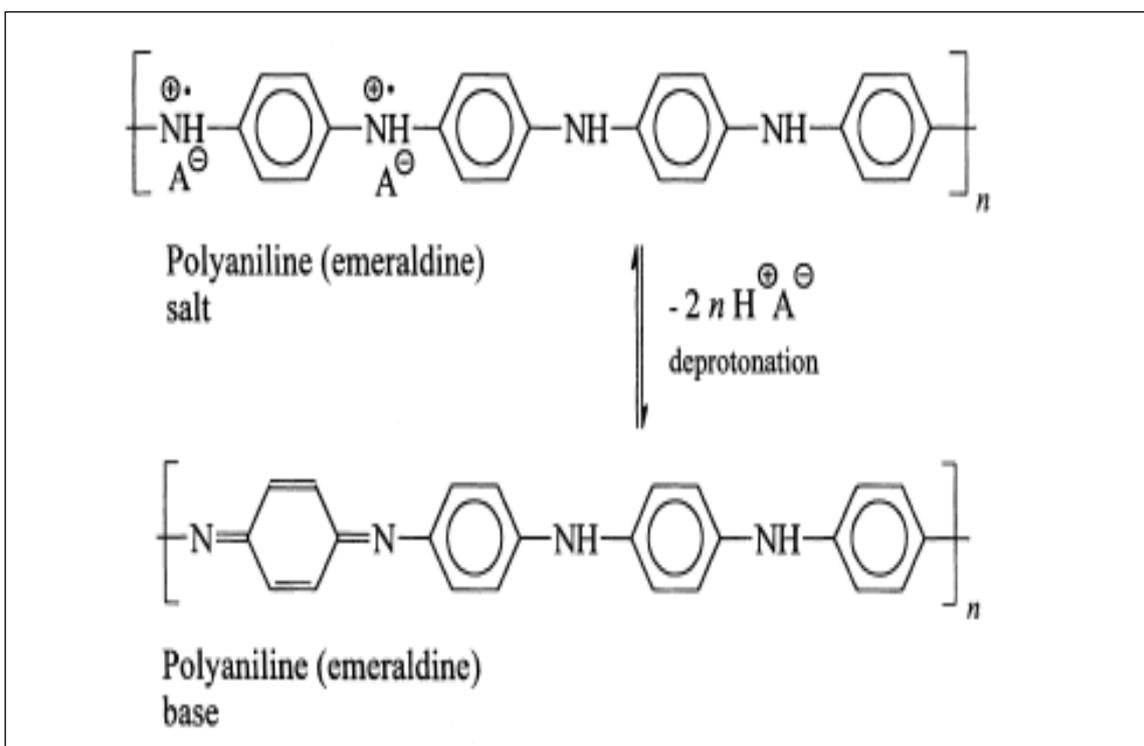
Figure: (1. 2) Polyaniline (emeraldine) salt is deprotonated in the alkaline medium to polyaniline (emeraldine) base. A− is an arbitrary anion, e.g., chloride [37].

Table (1.1): Types of Polyaniline Polymer [36].

Type of PANI	Color	Conductivity
Leucoemeraldine	white/clear	poor conductivity
Emeraldine	green or blue	good conductive if doped
Pernigraniline	blue/violet	poor conductivity

PANI -based materials have a wide range of applications by integrating a biodegradable into the matrix of PANI, a number of electrically conducting polymers many was set up the materials can be used in a variety of fields, including biomedical engineering, tissue engineering, and electronic sensors, conducting polymers are used in dental implants and targeted medication delivery, among other biomedical uses [38].

19.2 Okra Plant

Okra (bahmia)   also known as Lady's finger, is a monocotyledon herbaceous plant [39]. Okra plant fiber can be used not only as a filler from agricultural waste product but ideally to provide some reinforcement to the polymer matrix, where okra can be a source of polysaccharides, which can be used, with appropriate chemical grafting for polymer synthesis [40]. In this study, okra bioconductive polymer was selected as a model material which is used in many pharmaceutical, food, biomedical applications and scaffold for tissue engineering [41]. Polymeric compounds reinforced with plant fibers are characterized by their sustainability and light weight, as they are cost-effective, simple and less toxic compared to other polymeric compounds used for multi-nanoparticles attributed to metals [42,43].Okra is especially rich in bioactive compounds, particularly polysaccharides as mucilage form and phytochemicals [44, 45]. The compounds of okra plant are shown in table (1.2) [46,47] .

Table (1.2): The compounds of okra plant [46, 47].

Compounds of okra plant	Value	The repeating units of the okra plant mucilage
A-cellulose	60-70%	(1-2)-rhamnose
hemicellulose	15-20%	(1-4)-galacturonic acid residues with disaccharide side chains
lignin	5-10%	
pectin	3-5%	

1.9.3 Silver Nanoparticles

Silver (Ag) is a metal element, which is a soft, lustrous element possessing high electrical and thermal conductivity. It has been known extensively due to its medical and therapeutic benefits. It is practiced in many forms as coins, vessels, solutions, foil, sutures, and colloids [48,49]. Among the all metal nanoparticles, silver nanoparticle are an arch product from the field of nanotechnology which has gained boundless interests because of their unique properties such as chemical stability, good conductivity, catalytic and most important antibacterial, anti-viral, antifungal in addition to anti-inflammatory activities which can be incorporated into composite fibres, superconducting materials, food industry and electronic components [50]. A number of metallic silver particles can also be encapsulated in to the conductive polymer to form nanocomposites (NC). The NCs exhibit combination of properties like conductivity, electrochemical, catalytic and optical properties [51]. The incorporation of metal nanoparticles could effectively improve the electrical and optical properties of the polyaniline composites. The electrical conductivity such composites might also depend upon the molecular structure of the conductive polymer matrix (i.e., crystallinity). Since silver exhibits the highest electrical and thermal conductivities among all the metals [52], the combination of PANI with silver could yield functional materials having enhanced electrical properties. In this study, we explore the possibility of improving the conductivity of PANI by doping it with silver atoms. Ag properties are shown in Table (1.3).

Table (1.3): Properties of Ag nanoparticles [48, 53, 54].

Characteristic	Ag nanoparticles
atomic number	47
atomic mass	107.87
Purity	99.99%
Density	10.5 g/cm^3
color	black
Average particle size	20 nm
Morphology	Spherical
Refractive index	1.33 - 1.42
Surface area	18-22 m^2/g

1.10 Literature Survey

Sukitpaneenit. P et al. (2007) studied the DC electrical conductivity of the elastomeric PANI/NR composite fiber bundles was studied as a function of PANI concentration and electrically conducting elastomer fibers based on natural rubber (NR) and polyaniline (PANI) were fabricated. The morphology of the composite fibers was studied by scanning electron microscopy (SEM). The images show a combination of well dispersed, isolated PANI particles. PANI particles were inhomogeneously distributed in the NR matrix. The result indicate that the electrical conductivity of the fibers increased with the increasing PANI. Thus, the new composite fibers display a most desirable property matrix having properties that make them useful for applications [55].

Afzal. A. B et al. (2008) prepared PANI /Ag nanocomposites. Scanning electron microscopy showed a uniform distribution, with spherical and granular morphology for low concentration of Ag nanoparticles. FTIR spectra of the pure PANI as well PANI/Ag nanocomposite films are similar to the pure PANI, confirming the formation of PANI in all samples. They found that the Incorporation of silver nanoparticles in PANI reduces the charge trapping centres and increases the conducting channels [56].

Sezer.A et al. (2009) studied the refractive indices of polyaniline and polyaniline–silver nanocomposites. The intensity dependent refractive indices of polyaniline and polyaniline–silver nanocomposites are measured. While the material are not good candidates for all-optical switching, they do exhibit reversible processes of saturable and reverse saturable absorption at the same wavelength, a trait that makes them suitable materials for optical pulse compression and limiting. [57].

Gupta. K et al. (2010) studied the PANI/Ag nanocomposite has been synthesized successfully by the chemical oxidative polymerization of aniline with ammonium peroxydisulphate as an initiator in presence of silver nanoparticles. From the SEM image, it is observed that nanoparticles are well dispersed in the polyaniline matrix. The optical band gap of nanocomposite decreases with increasing content of silver nanoparticles. The electrical conductivity of polyaniline–silver nanocomposite increases with increase in silver nanoparticle content than that of pure polyaniline. This is a simple way by which optical and electrical properties of polyaniline may be enhanced by doping with suitable nanoparticles [58].

Thakur. V. K and Singha. (2010) prepared and characterizations of Natural Fibers/Polymer biocompositesusing Pine Needles (PN) reinforced polymers using Phenol-Formaldehyde (PF) as a novel polymer matrix has been

reported. the results indicate properties of Pine Needles reinforced Phenol-Formaldehyde (PF) resin-based composites have been found to be better than the parent polymeric Phenol-Formaldehyde resin and Pine Needles has immense scope in the production of natural fiber reinforced polymer composites having vast number of industrial applications [59].

Ghasemi-Mobarakeh.L et al. (2011) discussed the most commonly utilized conductive polymers, polypyrrole (PPy) and polyaniline (PANI), along with their design and modifications, thus making them suitable scaffolds for nerve tissue engineering.Electrical stimulation has been shown to enhance the nerve regeneration process and this consequently makes the use of electrically conductive polymers very attractive for the construction of scaffolds for nerve tissue engineering [60].

Razak. S. I. A et al. (2012) prepared newly modified biofibers made up of kenaf fibers (KF) and conducting PANI were successfully prepared via in situ polymerization. The PANI coated KF (KF/PANI) achieved new electronic properties, without sacrificing its mechanical properties and natural fiber characteristic. The mercerized KF/PANI exhibits polaronic transitions, enhanced DC conductivity, and better morphological characteristic as a result of the in situ PANI coating. Such electronically modified natural fibers could be suitable as green conducting fillers in composites to replace other synthetic fibers [61].

Safenaz.M.R et al. (2012) studied the electrical properties of nanocomposites (PANI/Ag) whereby a nanocomposite (PANI/Ag) was prepared by chemical oxidative polymerization of aniline monomer in the presence of nitric acid. Marked samples prepared (PANI/Ag)by FTIR and UV spectroscopy. The conductivity, dielectric permittivity $(\varepsilon)'$ and dielectric loss $(\varepsilon)''$ of the pure (Ag/PANI) nanocomposite and PANI were measured in the temperature range

from (303) to (723) K. It was found that the electrical conductivity of the nanocomposite (Ag/PANI) is higher than that of the (pure) PANI [62].

Kaveeta. P. J. (2012) prepared conducting PANI nanofibres combined with mupirocin. The prepared polymer was then tested for the antibacterial properties against various Gram positive and Gram negative bacteria such as Streptococcus pyogenes, Staphylococcus epidermidis, Staphylococcus aureus and Escherichia coli. Scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) were used to identify the chemical structure of the PANI nanofibres. The antibacterial properties were assessed by measuring the zones of inhibition. It was evident from these results that antimicrobial activity increased with increasing PANI and PANI combined with mupirocin (PANI-mupirocin) concentrations. This information might be useful to evaluate the potential use of nanostructured PANI in fabrics incorporated with antibacterial agents as a prophylactic use against bacterial skin infections [63].

Srilalitha.S, Jayaveera.K and Madhvendhra. S. (2013) presented a study on the effect of doping, temperature and energy gap on conductive polymers, where the bonded polymers showed the behavior of semiconductors and can be doped to give materials with high conductivity, where the effect of doping on the conductivity, the conduction mechanism, and the effect of the energy gap and temperature on polymers such as poly Pyrrole, polyaniline and polythiophene and their conductivity has been determined [64].

Chen.Y. H et al. (2013) comfortabled polymer-based dry electrodes are fabricated. The dry conductive polymer electrodes with the highest carbon black content had the impedance value approaching that of the gel electrodes. To characterize these electrodes, the impedance measurement and biopotential signals were performed. The influences of skin pretreatment techniques on impedance were studied, by applying gel of conventional ECG electrodes and abrasive gel on the skin. An important impedance reduction was obtained, when

applying the dry electrodes ECG/EEG systems. Based on the results the dry electrodes provide a high user comfort solution for high quality biopotential measurements, even on very hairy skin [65].

Silva. M. J. D et al. In (2014) they prepared a composite of natural rubber (NR) with PANI by doped PANI with NR in different concentrations. Films were obtained by the casting method and characterized by ultraviolet visible near-infrared (UV-Vis-NIR) spectroscopy and DC electrical conductivity measurements. The UV-vis-NIR spectrum showed that PANI remained doped in the composite, and this improved the mechanical and electrical proprieties of NR films and afforded them good thermal stability and in this case, conduction preferentially occurs by hopping [66].

Al-Oqla.F. M et al. (2015) studied conductive conductive polymer composites (CPCs) filled with natural fibers. The possibilities of utilizing natural fibers as fillers for ICPs to form natural fibers-conducting polymer composite materials have wide potentials in the modern industries. The unique characteristics such as electrical conductivity, mechanical strength, biodegradability and recyclability enabled them to be implemented in many novel and exciting applications including antennas, chemical sensors, tissue engineering, neural probes, biosensors, drug delivery, bio-actuators, fuel cells etc. On the other hand, it was reported that relatively short natural fibers could modify the dielectric response of the polymeric matrix [67].

Haruna.S, Aliyu.B. S and Bala.A. (2016) discussed the natural plant exudates and mucilages, their sources, properties and uses and the potential applications. Research in natural polymeric materials has witness growing interest and attention. This is attributable to a number of factors which include their relative abundance, low cost, biodegradable nontoxic, and ecofriendly profile. They are polymers that are mostly plant in origin with a vast applications. Polysaccharides hydrocolloids including gum and mucilage are abundant in nature and commonly found in many plants. These polysaccharides

constitute a structurally diverse class of biological macromolecules with a broad range of physicochemical properties which are widely used for various in pharmacy and medicine applications [68].

Ling. F. W and Abdulbari. H. A. (2017) studied the feasibility of three natural polymeric drag reducing additives in enhancing the flow in microchannel. was prepared by adding deionized water which acted as transported liquid after extracting from okra, aloevera and hibiscus leaves. The additives were then tested in custom made microchannel simulating human heart blood vessels. This work introduces an approach for flow enhancement in semi-clogged blood streams using natural polymers that could contribute as an alternative treatment for cardiovascular diseases [69].

Mohammadi. H et al. (2018) examined the physico mechanical parameters and antibacterial activity of CMC/okra mucilage (OM) blend films containing ZnO nanoparticles (NPs). Different proportions of CMC and okra mucilage were mixed and casted to posterior analysis of formed films. The more films were obtained by higher contents of okra mucilage and adding ZnO nanoparticles. Nanocomposite films presented antibacterial activity against tested bacteria. Films contained okra mucilage showed more antibacterial activity [70].

Karthikeyan.R et al. (2018) made Polyaniline and polyaniline composites of natural (coir fiber)/Synthetic fibers were synthesized by both insitu polymerization process and solution process. The natural fibers can be commonly used as ideal carriers of conducting polymer than synthetic fibers as they are abundantly present in nature, low cost, sustainable, renewable and recyclable. The electrical measurement shows increase in electrical conductivity of composites as compared to pure PANI. The SEM of PANI coated natural and synthetic fibers revealed that the coir fiber surface and glass fiber surface was completely coated with a uniform polyaniline layer which led to an electrical

conductivity quite similar to that of the pure polyaniline. The results obtained help in the development of conducting polymer composites for gas sensor applications [71].

Ramesan. M. T and Dilsha.K et al. (2019) studied the structural, morphological and electrical conductivity of polyaniline (PANI)/phenothiazine (PTZ) blend filled with different contents of copper sulphide (CuS) nanoparticles. The presence of CuS in the blend led to some changes in the position of peaks in the FTIR and UV spectrum. A granular morphology of the composite as compared to pure blend was confirmed by SEM images. DC conductivity of polymer blend increased with increase in CuS loading and showed maximum electrical properties. These results suggest that PANI/PTZ/CuS nanocomposites with improved electrical conductivities,seem to be promising materials for designing electrical or nano-electronic devices [72].

Zhang.Y et al. (2020) they designed of natural fiber/ PANI core-shell heterostructures with tunable and excellent electromagnetic shielding capability via a facile secondary doping strategy. natural bagasse fiber/ PANI (BF/PANI) core-shell composites were selected as the model heterostructures. It was demonstrated that both electrical characteristics and electromagnetic parameters of BF/PANI heterostructures can be easily tailored via a secondary doping strategy. calculations reveal that absorption are remarkably improved .Strong absorption behavior and power analysis confirm the absorption-dominated shielding mechanism. Taken together, these understandings will lead to a rational design of PANI-based heterostructures to achieve ad justable, superior shielding capabilities [73].

Aghazadeh.H et al. (2021) fabricated PANI coated stainless steel (SS) electrodes using electrochemical polymerization on the SS surface. The SEM

images showed the formation of a nanoporous PANI-coating on the SS electrodes. measurements on a skin model demonstrated a significantly lower contact impedance for the PANI-coated electrodes compared to bare SS electrodes. Electrodes are essential devices for monitoring of the biopotential such as electroencephalography (EEG) and electrocardiography (ECG) [74].

De Menezes.L. R et al. (2021) studied the development of new materials for obtaining ophthalmic lenses due to the great necessity of corrective lenses uses. The use of nanoparticles in polymer nanocomposites can generate different properties in the final material, evaluate the optical properties of systems with poly (methyl methacrylate) and oxide nanoparticles (silica, zinc, zirconium and titanium). The systems were characterized in terms of their optical behaviors refractive index and transmittance behaviors.The results showed that silica-based systems tended to have greater transpar ency but generate lower refractive indexes. Systems containing zirconia, titanium, and zinc have a higher refractive index and smaller transparency than systems containing silica. The combination of the nanoparticles also led to a better dispersion, reducing the impact on the color system and improve transparency [75].

Atta. A et al. (2022) fabricated   novel flexible polymer composite films composed of PANI, Ag nanoparticles (Ag-NPs), and methylcellulose for portable and wearable electronic devices. The MC/PANI/Ag composite films are characterized using scanning electron microscope (SEM) and Fourier transform infrared (FTIR) techniques.The infrared spectroscopy, confirmed the successful fabrication of the MC/PANI/Ag composite films. The SEM images have indicated a homogenous loading and dispersion of Ag-NPs into the MC film. The electrical conductivity has increased for MC/PANI/Ag-NPs. These results confirmed that there are significant modifications on the fabricated films,

which open the road for utilizing the MC/PANI/AgNPs as flexible films for wide range of electronic applications [76].

Gao.M et al. (2022) studied graphite (G) powder was used as the substrate for the growth of aniline by in-situ polymerization to get a PANI/G composite as an electrode material. The results showed that the electrical conductivity of the PANI/G composite was improved greatly, which promotes this cheap composite to be used in the electrochemistry industry widely [77].

1.8 The Aims of The Study

The principle aim of the current study is to prepare bio-composite based polymer from polyaniline and okra plant waste to apply for some medical applications. During the research some objective should be achieved:

1- Study the structural properties of the synthesis sample.
2- Study the optical properties of the biopolymer composite.
3- Study the electrical characterization of the polyaniline modifidokra plant.
4- Study the antibacterial activity towards of the polyaniline modified okra plant.
5- Investigate the electrode efficiency towards of the polyaniline modified okra plant.
6- Study the application of eye glasses towards of the polyaniline modified okra plant.
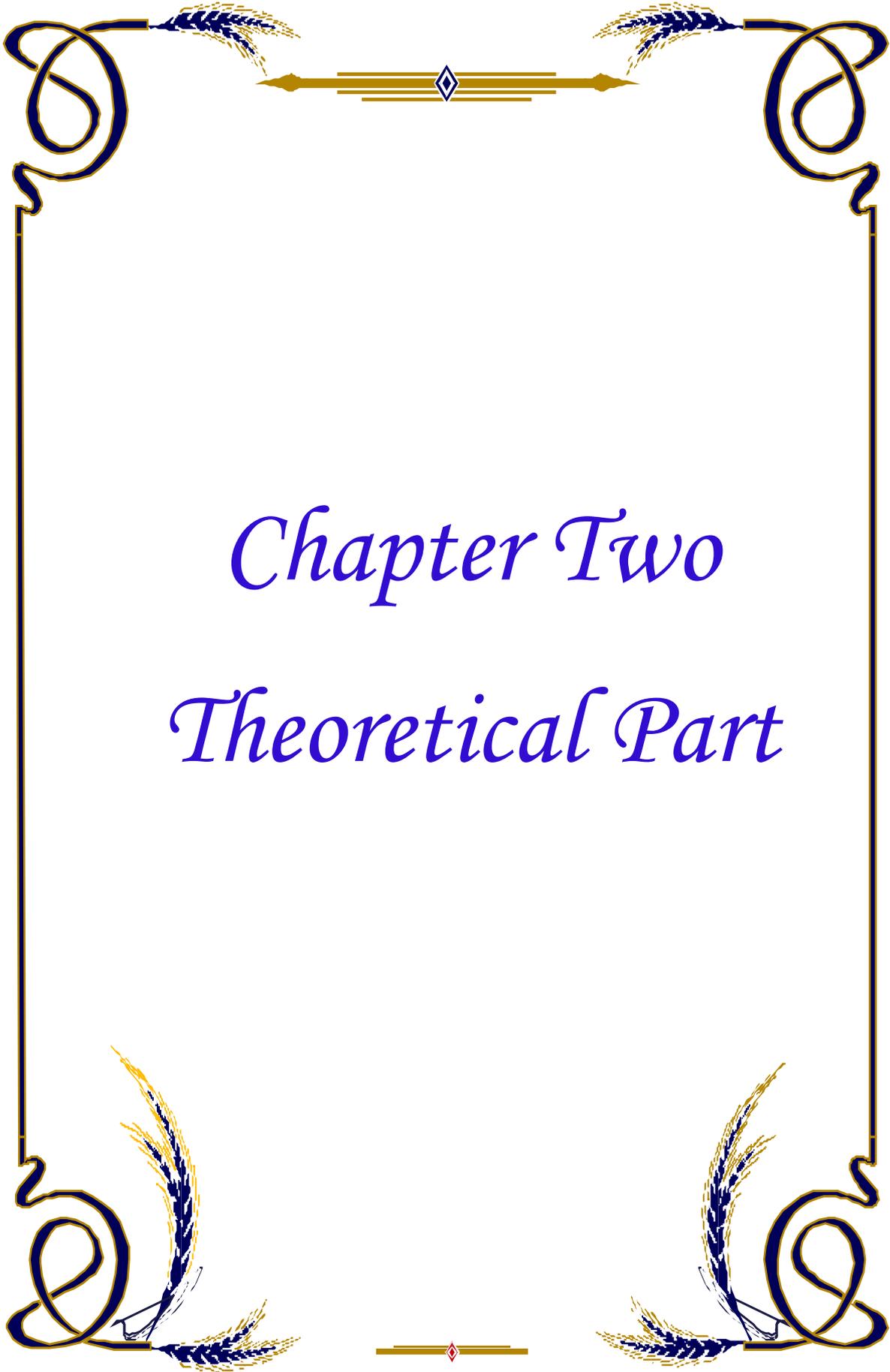
Chapter Two

Theoretical Part

2.1 Introduction

This chapter includes general description of the theoretical part of the current study, relationships, and the laws used to interpret the obtained results.

2.2 The Optical Properties

The optical properties of materials are properties that focus on the interaction between electromagnetic radiation or light with an object, which include absorption, polarization, reflection and scattering effects [78].

2.2.1 The Absorbance (A)

Absorption can be defined as the ratio between the absorbed light intensity (I_A) by material and the intensity of incident light (I_o) [79].

$$A = \log (I_o / I) \tag{2-1}$$

2.2 2. Transmittance (T)

The transmittance is given by the ratio of the intensity of the transmitting rays (I_T) through the film to the intensity of the incident rays (I_o) on it as follows [80]:

$$T = I_T / I_o \tag{2-2}$$

The reflectance can be obtained from absorption and transmission spectra in accordance with the law of conservation of energy by the following relation [81]:

$$R + T + A = 1 \tag{2-3}$$

2.2.3 Absorption Coefficient (α)

The absorption coefficient of the material is a function of photon energy and energy gap [82]. Absorption coefficient (α) is defined as the ability of a material to absorb the light of a given wavelength[83].

The absorption coefficient at a fundamental absorption edge can be written as [84]:

$$\alpha = 2.303A/t \qquad\qquad\qquad (2\text{-}4)$$

where A: absorbance, t: thickness.

2.2.4 The Refractive Index and Extinction Coefficient

Refractive index of the material is the ratio of the velocity of light in a

vacuum to the velocity of the light in the sample [85]:

$$n = c/v \qquad\qquad\qquad (2\text{-}5)$$

Where c: is the velocity of the light in vacuum and v: is the velocity of the light in the sample.

$$n^* = n - ik \qquad\qquad\qquad (2\text{-}6)$$

n^* : is the complex refractive index, n : is a real part of the refractive index and k : is an imaginary part of the refractive index (extinction coefficient).

The relation between absorption coefficient and the extinction

coefficient (k) is [86]:

$$k = \alpha\lambda/4\pi \qquad\qquad\qquad (2\text{-}7)$$

The refractive index can be expressed through the following equation [87]:

$$n = \left[\left(\frac{1+R}{1-R}\right)^2 - (k_o^2 + 1)\right]^{\frac{1}{2}} + \frac{1+R}{1-R} \qquad\qquad (2-8)$$

Since the value of extinction coefficient (k) is very small, then the equation (2-8) will become as follows [84]:

$$n = 1 + (R)^{1/2}/1 - (R)^{1/2} \qquad\qquad\qquad (2\text{-}9)$$

2.2.5 The Dielectric Constant (ε)

The real half of the dielectric constant describes how much it slows down the speed of light in the sample, whereas the imaginary part describes how a dielectric absorbs energy from an electric field owing to dipole movement. found the real and imaginary parts of the dielectric constant [87]:

$$\varepsilon = \varepsilon_r - i\varepsilon_i \tag{2-10}$$

$$\varepsilon = (n*)^2 \tag{2-11}$$

$$(n- i\,k)^2 = \varepsilon^r - i\,\varepsilon^i$$

$$\varepsilon = (n^2 - k^2) - i\,(2\,n\,k) \tag{2-12}$$

The dielectric coefficient (ε) may be determined from the refractive index (n) by multiplying the complex dielectric coefficient (ε) by the complex refractive index (n*) from equation (2-10) and (2-12) real and imaginary complex dielectric coefficient can be written as in following equation [88]:

$$\varepsilon_r = (n^2 - k^2) \tag{2-13}$$

$$\varepsilon_i = (2\,n\,k) \tag{2-14}$$

2.2.6 Optical Conductivity (σ_{opt})

The optical conductivity (σ_{opt}) depends directly on the refractive index (n) and extinction coefficient (k) by the following relation [88] :

$$\sigma_{opt} = 2nk\omega\varepsilon_o \tag{2-15}$$

Where ω: is the angular frequency, ε_o: is the permittivity in the free space. By substituting (2-7) in (2-15) with simplified, the optical conductivity can be expressed by the following equation:

$$\sigma_{opt} = \frac{\alpha\,n\,c}{4\pi} \tag{2-16}$$

2.3 Electronic Transitions

There are two types of electronic transfers, direct electronic transmissions and indirect electronic transmissions.

2.3.1 Direct Transitions

The electron moves from the valence band (V.B) to the conduction band (C.B) at the same point in the wave vector space within the condition ($\Delta k = 0$) when it absorbs a photon whose energy is greater or equal to the energy of the gap (hv = E_g) to occupy a place in the conduction beam, where the law of conservation of energy and momentum is fulfilled and there two types of direct transfers. When the transition occurs between the direct points of the top of the equivalence beam and the bottom of the conduction beam, it is called transition direct allowed as shown in Figure (2.3a). Adjacent to the top of the valence band to a point adjacent to the bottom of the conduction band it is called forbidden direct transmission as shown in figure (2.3b) [62].

2.3.2 Indirect Transitions

In indirect transitions the conduction band bottom and the valence band peak are in different regions of the wave space, meaning that the value of the wave vector of the electron before and after the transition is unequal. ($\Delta k{\neq}0$) and this type of transition occurs with the help of the phonon in order to conserve the momentum resulting from the change of the wave vector of the electron. There are two types of indirect transfers, when the transfers between points are indirect direction to the highest point of the valence band and the lowest point of the conduction band located in different regions for k space, it is called the indirect transition allowed as shown in figure (2.3c). But if the transition is between adjacent points to the highest and lowest point in the equivalence band and the conduction band then it is called the forbidden indirect

transition as shown in figure (2.3d) [62]. The direct and indirect transitions can be calculated by using the Tauc's relation [89]:

$$\alpha h\nu = (h\nu - E_g)^r \tag{2-17}$$

Where, ν: is the frequency, h: is Planck's constant, E_g : is the energy gap between the valence band and the conduction band and r: is exponential constant, its value depends on the type of transition as listed in table (2.1).
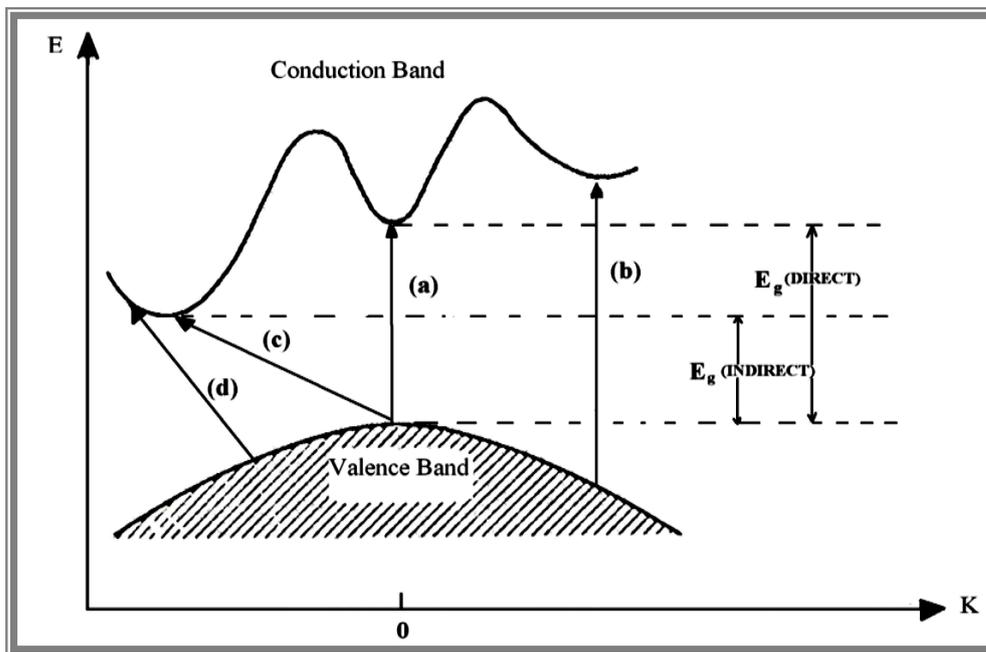
Figure (2.3): The transition process [90]

(a) Allowed direct transition.   (c) Allowed indirect transition.

(b) Forbidden direct transition.   (d) Forbidden indirect transition.

Table (2.1) Values of optical transition mode [62].

r	Transition mode
1/2	direct allowed
3/2	direct forbidden
2	indirect allowed
3	indirect forbidden

2.4 Fundamental Absorption Edge

Fundamental absorption refers to band-to-band or excitation transitions (i.e., the excitation of an electron from the valence band to the conduction band), and it is manifested by a rapid rise in absorption. Three types of absorption areas have been classified [91].

2.4.1 High Absorption Region

Figure (2.4) shows the region the character of the electron transitions is described as direct in section (A). The coefficient of absorption has a magnitude greater than or equal to (10^4 cm^{-1}) [92].

2.4.2 Exponential Region

The transition from extended levels in the valence band to localized levels in the conduction band is described in section (B) in figure (2.4). From localized levels in the top valence band to extended levels in the bottom conduction band. The value of the coefficient of absorption between ($1<\alpha<10^4$) cm^{-1}[96].

2.4.3 Low Absorption Region

In this region the absorption coefficient (α) is quite low, around ($\alpha<1$cm^{-1}). As illustrated in figure (2.4) in section (C), the transitions of electrons in this region attribute to density of states as a result of structural flaws [93].
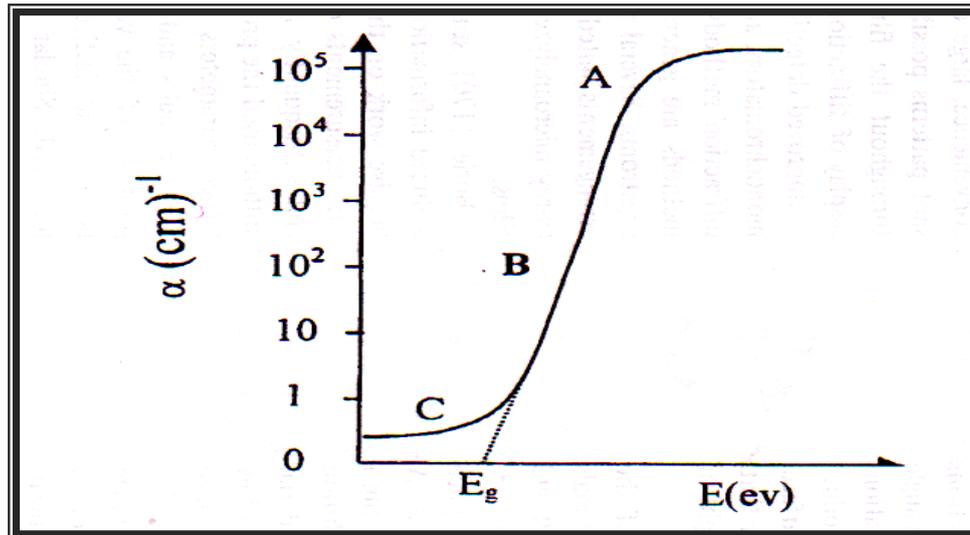
Figure (2.4): The variation of absorption edge with absorption regions [93].

2.5 The D.C Electrical Conductivity

The electrical properties of polymer composites reinforced with natural fibers are very important, due to their uniqueness, inorganic and electrical insulators have been replaced by polymers to meet specific needs [4]. Studied on the electrical properties of polymers have included significant interest in their applications in electronic and optical devices. Electrical conductivity in conductive polymers has been studied extensively to understand the nature of charge transfer in polymers [94]. The D.C electrical conductivity studies aim to understand the numbers of types of charge carriers, their source and how they are transported through the material [95].

The electrical conductivity in materials depends mainly on the temperature, which increases with increasing temperature in order to increase the number of charge carriers [96].The electrical conductivity is given as a function of temperature by using Arrhenius equation [97]:

$$\sigma_{DC} = \sigma_0 \exp\left(\frac{-E_a}{k_B T}\right) \tag{2-18}$$

σ_{DC}: electrical conductivity at T temperature.

σ_0: electrical conductivity at absolute zero of temperature.

K_B: Boltzmann constant.

E_a: activation energy.

As for the specific resistance of the material, it can be calculated using the following equation [98]:

$$\rho = R.A_o/L \qquad\qquad (2-19)$$

where:

R: the resistance of the sample.

A_o: the cross-sectional area of the sample.

L: the length of the distance between the electrodes.

Thus, the electrical conductivity of the prepared samples can be found through the following relationship

$$\sigma_{Dc} = 1/\rho \qquad\qquad (2-20)$$

2.6 Medical Applications of Conducting Polymers

2.6.1 Antibacterial Activity

The staining of bacterial cells on glass slides for visibility and characterization is a basic laboratory technique taught in general biology and microbiology courses. The gram stain is a typical method for distinguishing between bacterial species based on the chemical makeup of their cell walls. The staining technique includes using crystal violet as a primary stain, then adding gram's iodine as a mordant, decolorizing with an organic solvent such ethanol, then counterstaining with safranin. Following the procedure, gram positive bacteria, which are more resistant to decolorization, appear purpl in color while gram negative bacteria, which are more sensitive to decolorization, appear pink [99] as shown in figure (2.5) [100].
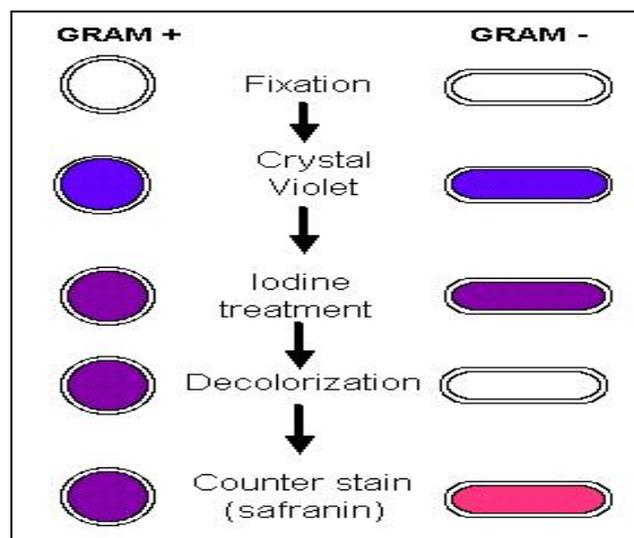
Figure (2.5)꞉ **The steps in gram staining [100].**

The presence or absence of bacterial illness can be determined via Gram stain interpretation, which can help guide first antibiotic treatment. Gram stain also offers information about the immunological response of the host and the specimen's quality. The color, size, form, and arrangement of bacteria can be seen in a well-prepared sample, allowing cellular morphology to further divide bacteria into four major groups. Cocci are spherical or oval, bacilli are rod like or cylindrical, vibrios are comma shaped or curved like and spirochetes are flexible [101].

Okra is one of the most important mucilaginous vegetable. It is especially rich in bioactive compounds, particularly polysaccharides as mucilage form and phytochemicals. Okra antibacterial activity may be attribute to polyphenolic compounds in okra extracts by disrupting cell membranes and destroying cell walls, causing intracellular components leakage and in some cases cell lysis. This study provides evidence of a potential application of okra mucilage as a novel functional ingredient, particularly being as a natural antibacterial [102].

Nanoparticles (Np$_s$) their active surface area, chemical reactivity and biological activity are often different from larger size particles [103]. NPs interact with the bacterial cell's basic components, such as changes in cell

membrane permeability, DNA, ribosomes and enzymes, leading to oxidative stress, enzyme inhibition, protein deactivation and changes in gene expression [104].

2.6.2 Electrodes

Polymer electrolytes (PEs) were introduced for the first time by Fenton et al. in 1973 and their technological significance on large-scale applications came to market in early 1980. Over the past three decades, a major focus on developing new PEs has been observed because of their wide uses in electrochemical supercapacitors, storage and conversion systems. PEs are membranes that consist of incorporated dissolved salts in polymer matrices. These almost solid without solvent systems possess ionic conduction property; therefore, they have extensively been used in a number of electrochemical devices, for example, rechargeable batteries and solid-state batteries. The synthetic and natural polymer-based electrolytes have been increasingly focused in terms of electrical and optical properties because of legibility to be utilized [105].

manufacturing of conductive polymer, metrics to assess their performance as electrodes, in the acquisition of critical biopotential signals for routine monitoring assessment of cardiac (electrocardiography, ECG), neural (electroencephalography, EEG), muscular (electromyography, EMG) [106,107]. Electrodes of ECG, EEG and EMG Illustrates in figure (2.6).
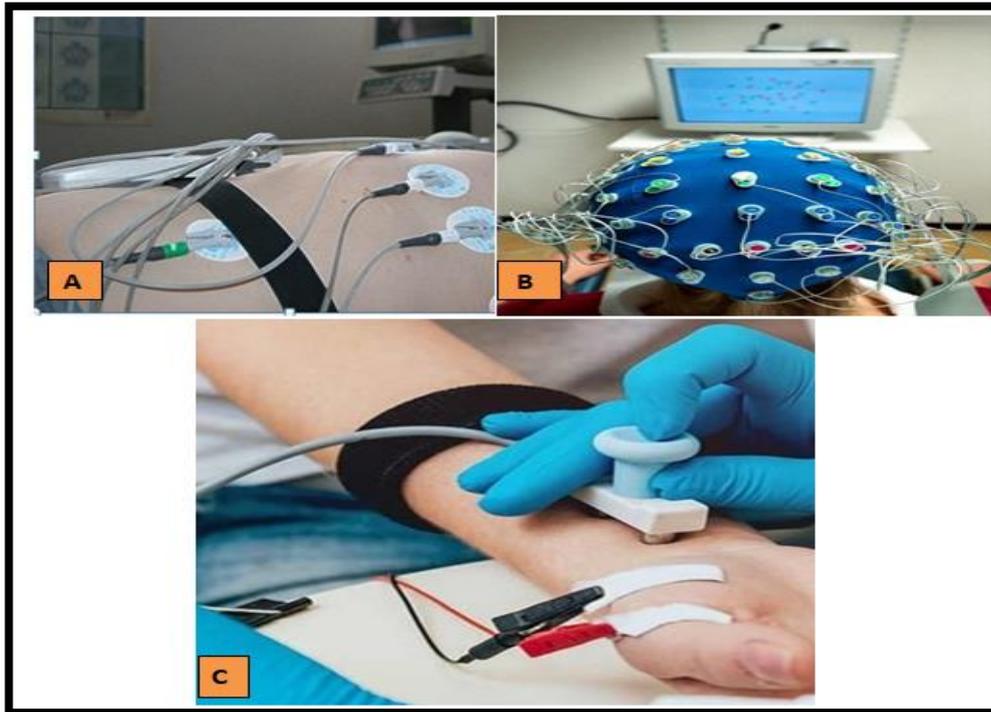
**Figure (2.6): Image of (A)(electroencephalography, EEG) (B)
(electrocardiography, ECG) (C) (electromyography, EMG).**

2.6.3 Eyeglass Lenses

Eyeglass lenses are used primarily to correct eye defects of eyeglass users. However, apart from the correction itself, several factors are important, the quality of the lens determines the resistance to abrasion it determines the durability of the product and its longest possible use. Another important factor is the transmission of light. A good lens should transmit the maximum amount of light in the visible range and completely cut off radiation from the ultraviolet range, which is extremely harmful to the optical system of the eye. The cornea of the human eye absorbs almost completely electromagnetic radiation with a wavelength less than 280 nm. Waves with a length of 300 to 370 nm are mostly retained by the lens of the eye. In contrast, radiation from the 380 - 400 nm range reaches the retina. The greatest risk associated with exposure to this type of radiation is the development of Age Macular Degeneration (AMD) leading to loss of vision [108]. The first eyeglass lenses were made of mineral materials

their advantage was and still is high abrasion resistance. Mineral materials such as quartz. Lenses made of mineral materials however are not without flaws. The main shortcomings of this kind of lenses are the susceptibility to cracking and a relatively large mass causing cumbersome use. The answer to problems arising from the use of mineral lenses are organic lenses. Polymeric materials are actively used in new optical devices. Modern advances in the synthesis, manufacture, and processing of polymers make it possible to use them in development of eyeglasses using components from polymer optical materials [108,109].

2.6.4 Gradient-Refractive Index Lens (GRIN)

Today's applications are driving a need for smaller, lighter optical devices without any sacrifice in image quality. One way to achieve these goals is to pack more functionality into each lens element. This has renewed the interest in GRIN materials, which are a natural choice for compact optical design. The basic building block of the GRIN lens is a set of transparent nanolayered polymer films, each with a specified refractive index. These layers are combined to produces a film with thousands of alternating layers of two polymers. The use index gradients to enhance focusing power, correct aberrations, and reduce the number of components needed for an effective optical system [110].
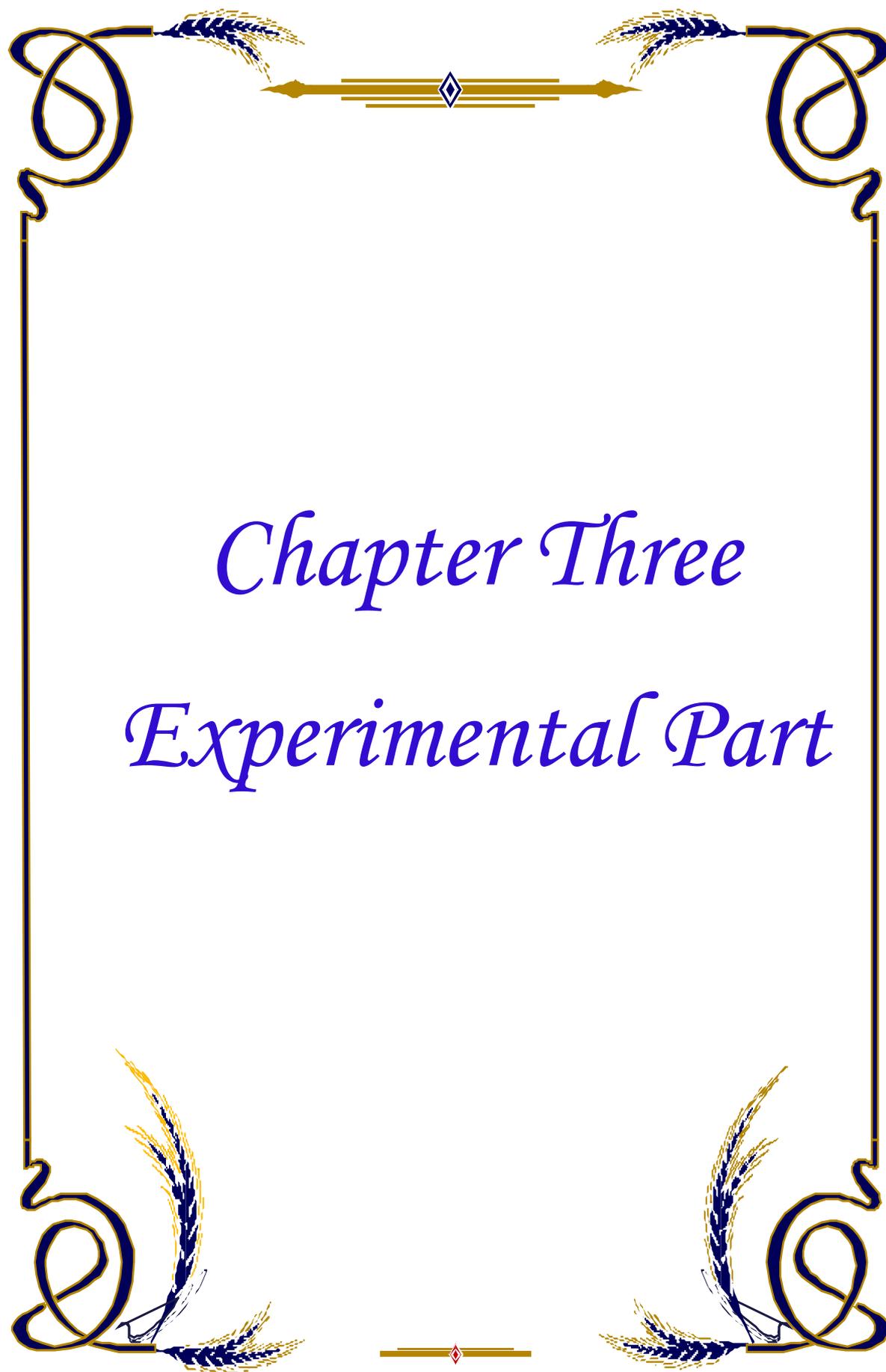
Chapter Three

Experimental Part

3.1 Introduction

This chapter includes the preparation process, devices and measurement techniques. A general description of materials (PANI, okra plant and silver nanoparticals) used in this work are given by Scanning electron microscopy (SEM), Atomic Force Microscope (AFM), Fourier Transform Infrared Radiation (FTIR), optical properties measurements, measurement of D.C electrical conductivity and includes some diagrams of electrical circuits.

3.2 The Materials Used in This Work

3.2.1 Polymer

Polyaniline (PANI):

Used as powder form and can be obtained from local markets with high purity (99.8%).

3.2.2 Natural Fiber

Plant fiber are used in this work is okra plant and can be obtained from local markets.

3.2.3 Additive Nanoparticles

Silver Nanoparticles

Used as powder with avarge particle size (20) nm from US Research Nanomaterial, Inc company and high purity (99.99%).

3.3 Samples Preparation

1- prepared the solution of 1 g of PANI in 50 ml chloroform, has been prepared then stirred with a magnetic stirrer for 12 hour to make it homogenous.

2- To prepare okra plant waste wash it thoroughly with water, cut into pieces, and soak it overnight in distilled water. After that, the mucilage was extracted by filtering it through cloth and the extracted mucilage was

dried in a 40° C oven to get apowder then 1g of okra powder is dissolved in 50 ml of chloroform and stirred with a magnetic stirrer for 60 minutes to get okra solution.

3- (PANI/okra plant waste) blend prepared by mix the two solutions PANI and okra plant waste for 60 minutes with a magnetic stirrer to make a homogenous solution.

4- The (PANI/okra plant waste/ Ag) nanocomposites are prepared by added Ag nanoparticles for the purpose of doping different proportions (0.5%, 1%, 1.5 % and 2%) to (PANI/okra plant waste) blend and mixed with a magnetic stirrer for 30 minutes to make the mixture more homogenous after that the films were prepared by spin coating method. The stages of the experimental work and procedure are illustrated in figure (3.1).
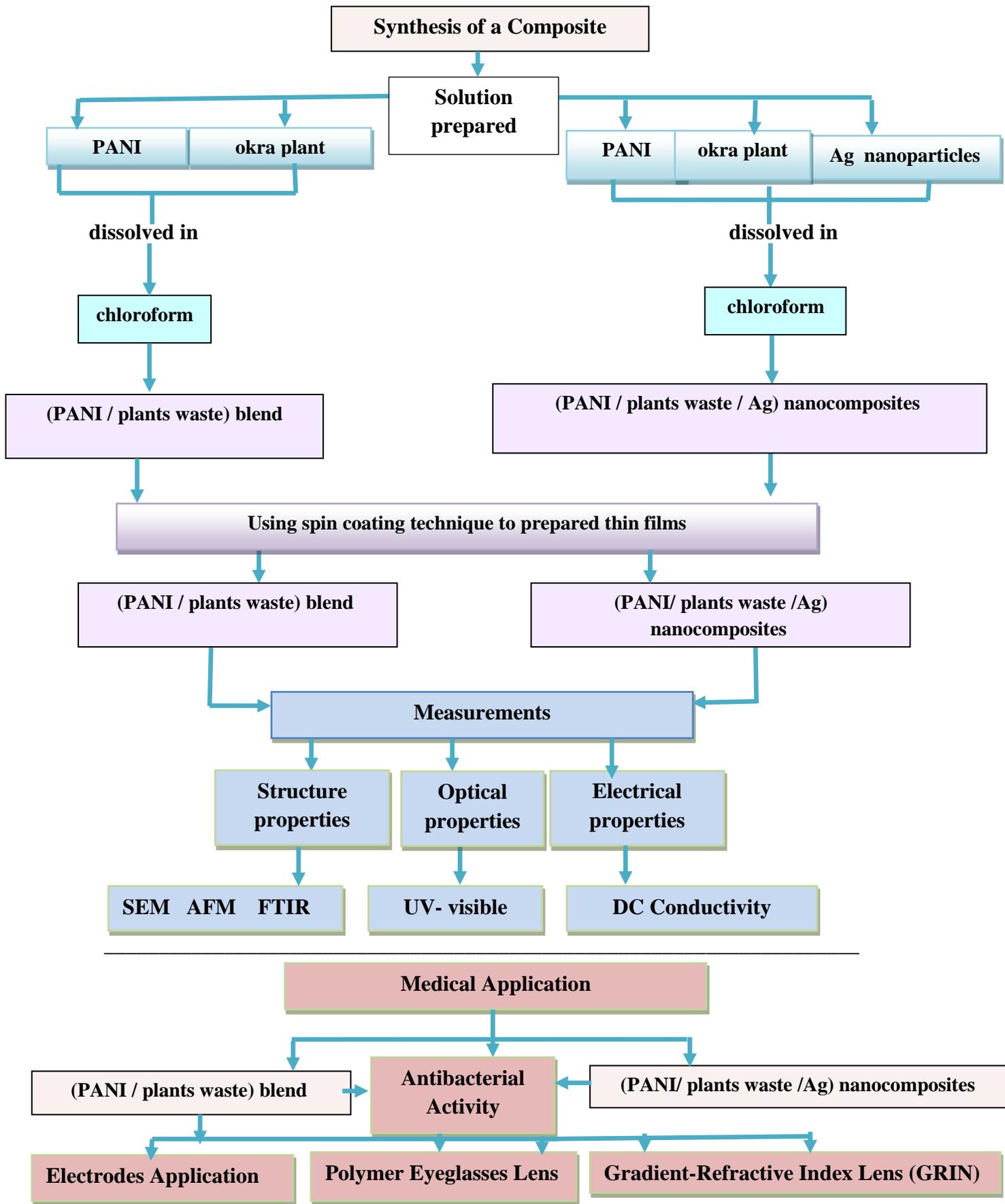
Figure (3.1): Schematic diagram of the experimental work.

3.4 Measurement of the Structural Properties

The structural properties of the thin films prepared by spin coating in this work by carrying out some measurements that are summarized as follows.

3.4.1 Scanning Electron Microscope (SEM)

Scanning electron microscopy (SEM) used in this research. SEM uses a concentrated electron beam to scan a surface and create a picture. When electrons in the beam interact with the specimen, they produce a variety of signals that can be used to inspect the morphology of the surface for polymeric blend and nanocomposite specimens with a very high magnification capacity and obtain information about the surface topography. The Schematic diagram for Scanning electron microscopy is shown in figure (3.2).
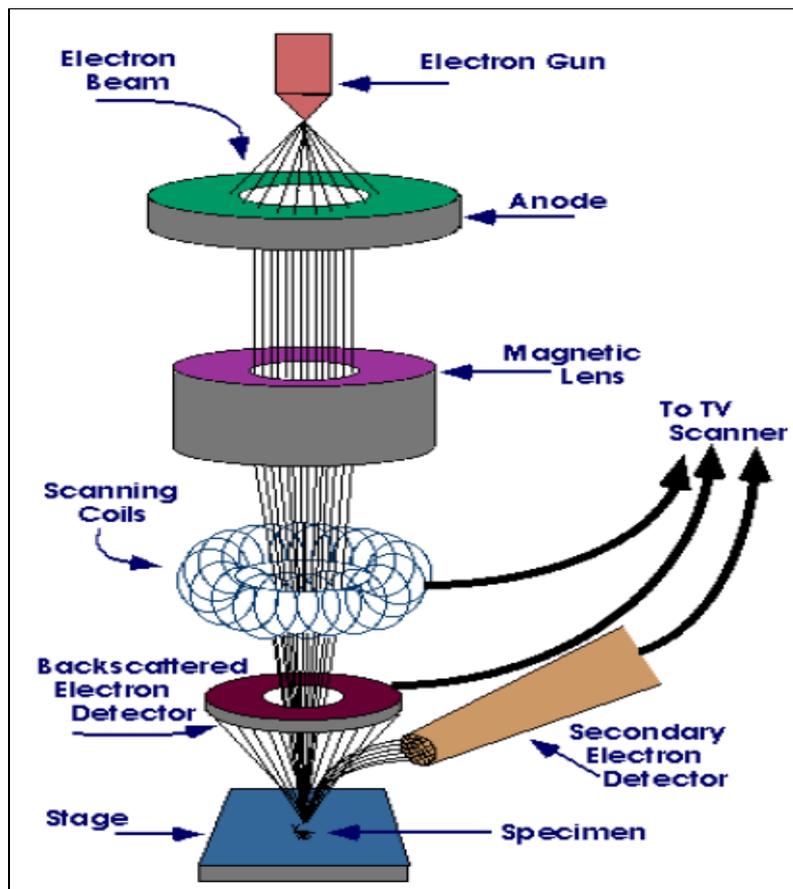
Figure (3.2): Scanning Electron Microscope (SEM).

3.4.2 Atomic Force Microscope (AFM)

Atomic force microscope (AFM) is a high-precision technique type of scanning probe microscopy, through which to investigate examine at the surface of a very high accuracy. AFM is used for surface imaging technology to obtain information about the surface morphology of the film, such as, distributing grains and knowing the sizes of the particles that make up the materials and the extent of their homogeneity and also the surface roughness. Many of the materials are identified by this technique, including polymers, semiconductors, metals and composites. The topography of the scanned surface is determined and drawn according to the movement of the reflected laser beam. This analysis was carried out in Iran, as shown in the following figure (3.3).
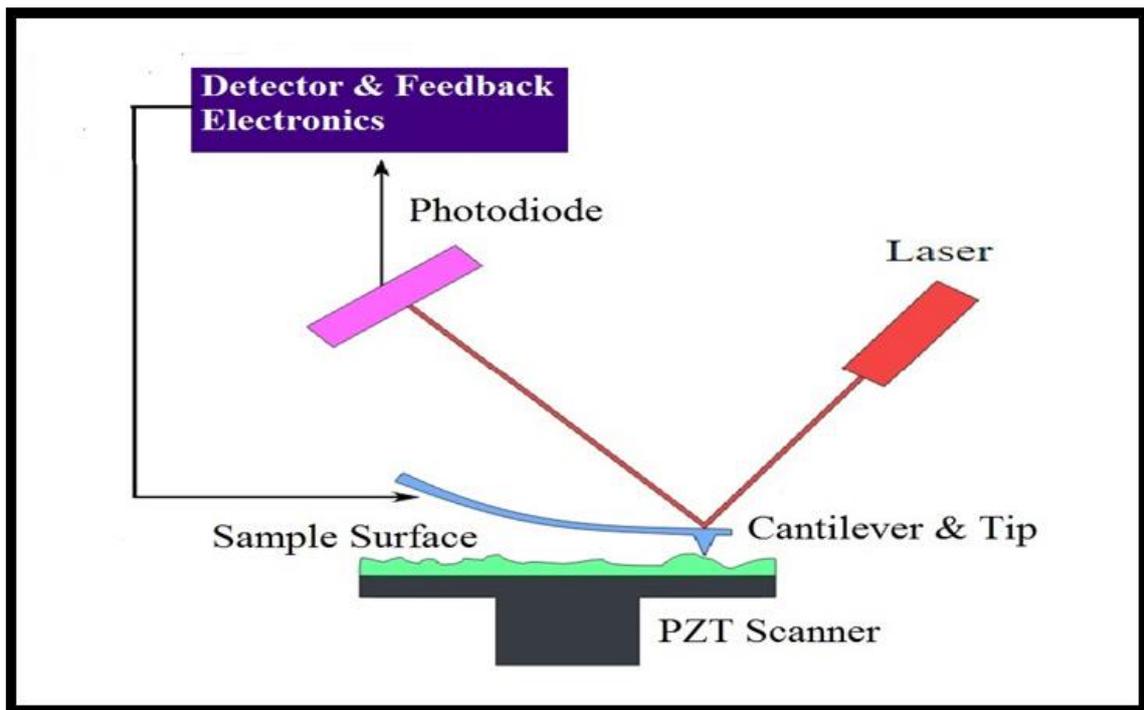
Figure (3.3): Diagram for atomic forces microscope (AFM).

3.4.3 Optical Measurements

Figure (3.4) displays a schematic diagram of optical circuit of an UV-Vis spectrophotometer with a dual-beam function. The broad range radiation absorption spectra were recorded by using UV/1800/Shimadzu spectrophotometer in range of wavelength (200-1100) nm. This instrument is present in the Polymer laboratory of Physics Department, College of Science, University of Babylon.
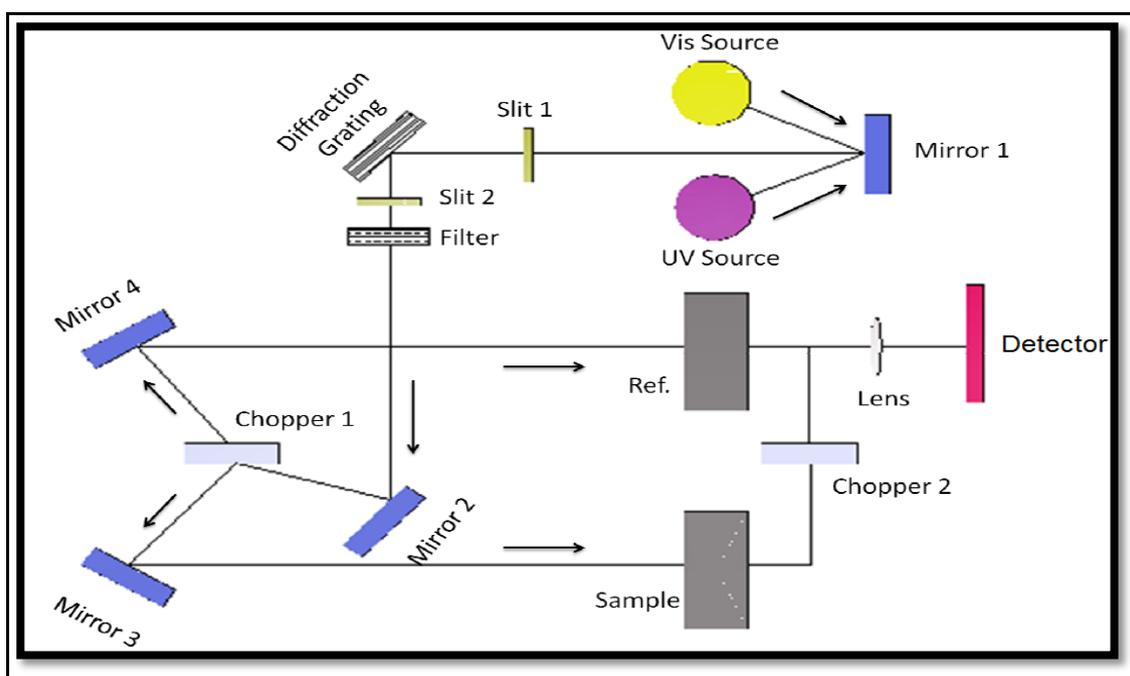
Figure (3.4): Schematic of an optical circuit for an UV-Vis spectrophotometer (dual-beam function).

3.5 Preparation of Masks and Electrodes Deposition

Types of masks have been made for electrical and measurements. The masks are shown in figure (3.5) from aluminum foil sheets in order to get the desired shape of electrodes. These masks have the same dimensions of the substrate and were exactly attached and fixed to cover the substrate after being cleaned. The gold electrodes deposited on the surface of films prepared by using (DC -SPUTTERING), this instrument is present in the laboratory of thin films in the University of Babylon/College of Science/Department of Physics.
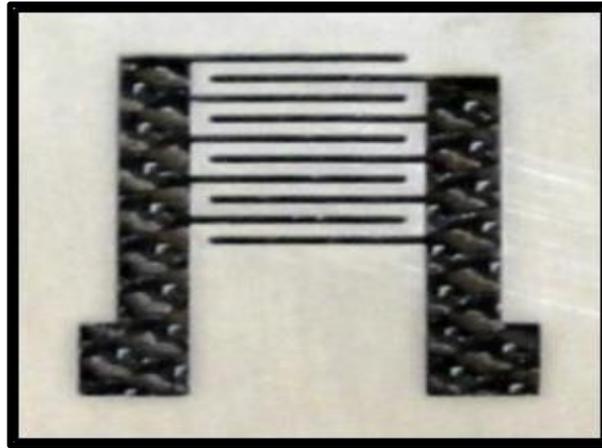
Fig (3.5): Interdigitated finger electrode masks of D.C conductivity measurement.

3.6 Measurements of D.C. Electrical Properties

The D.C electrical conductivity of the films prepared is measured in the University of Babylon/College of Science/Department of Physics-Thin Films Laboratory by using the circuit for measuring D.C conductivity shown in figure (3.6), which consists of an electrical oven type (Memmert Lab Oven UFB 400,400W) and a Keithly model 2400, with the sample placed inside the oven and the temperature in the range (20-110) ^{o}C an increase (10) ^{o}C in each step using the electrical circuit then the electrical resistance was measured directly for all steps using a digital electrometer.
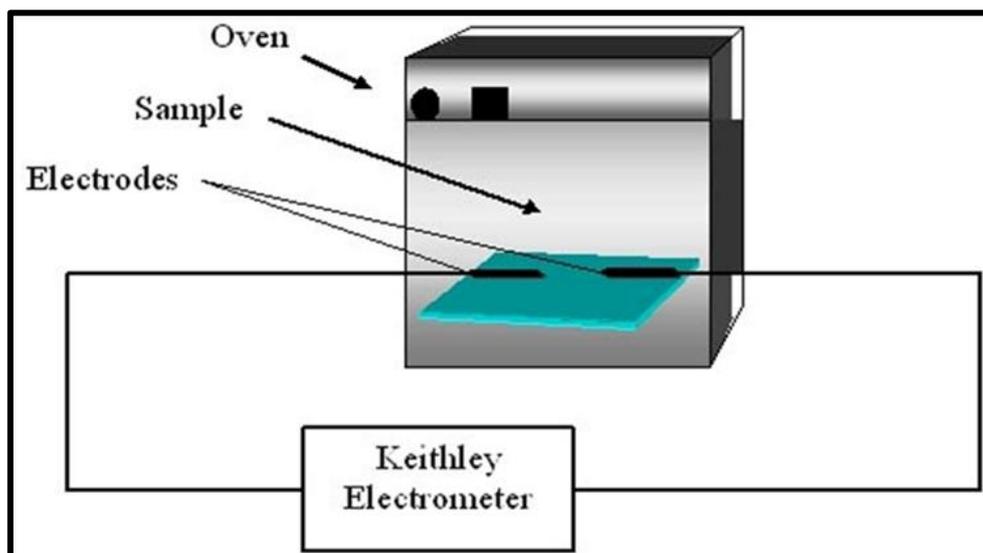
Figure (3.6): Circuit for measuring D.C conductivity.

3.7 Antibacterial Activity Application Measurements

3.7.1 Preparation of Media

Mueller Hinton agar (MHA) media were prepared in distilled water and all components were dissolved by the microwave oven to dissolve fully. The medium were sterilized at (121)°C for (15) minutes and then stored under a sterile environment at room temperature for subsequent use.

3.7.2 Preparation of Bacteria

The first step of the present work was obtaining different microorganism and testing the resistance of (PANI/plants waste) blends and (PANI/ plants waste/Ag) nanocomposites films against two microorganisms namely Staphylococcus epidermidis as gram positive bacteria and Klebsiella pneumoniae as gram negative bacteria Figure (3.7) shows images for the tested bacteria types.
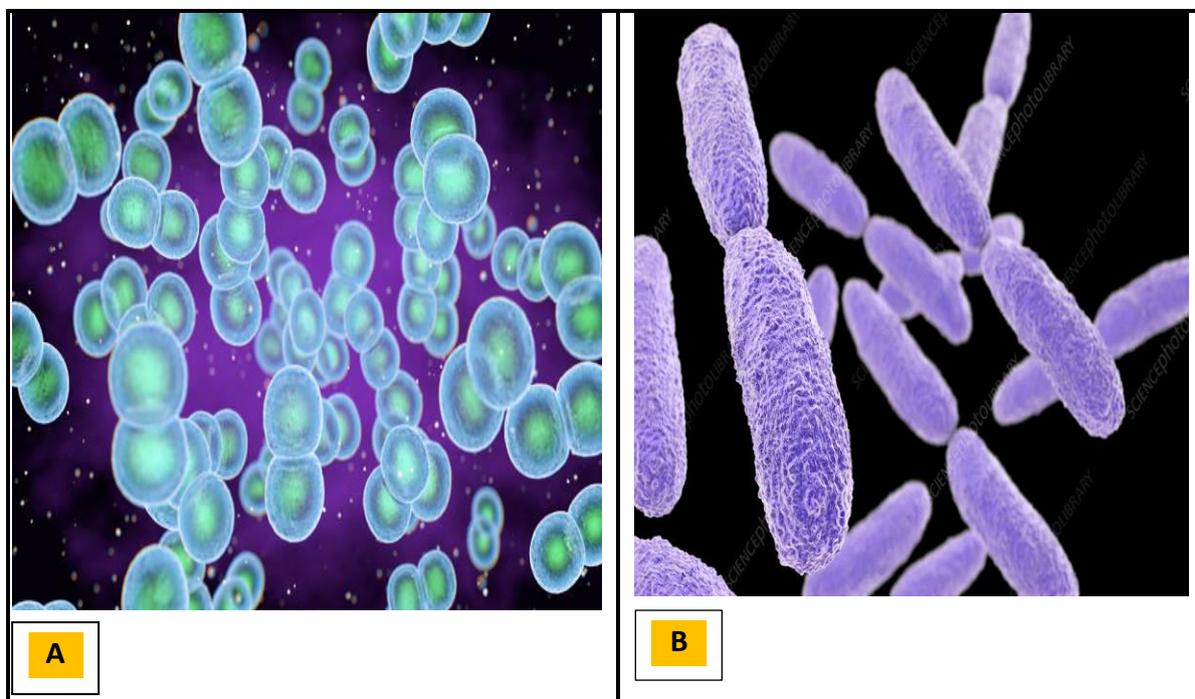
Figure (3.7): Images for (A) Staphylococcus epidermidis[111] (B)Klebsiella pneumonia [112].

3.7. 3 Preparation of McFarland 0.5 Standard

The (PANI/plants waste) blends and (PANI/ plants waste/Ag) nanocomposites films were tested to evaluate their antibacterial activity against two different types of clinical bacteria Staphylococcus epidermidis as gram positive bacteria and Klebsiella pneumoniae as gram negative bacteria by using well diffusion method. Standardized suspension of each tested bacteria ($5x10^8$ CFU/mL) by Dense Check standard (0.5) was swabbed separately onto MHA plates using sterile cotton swabs. The accuracy of bacterial suspension was confirmed using a spectrophotometer. Sterile MHA was used and measured at a wavelength of (625) nm as a blank solution for comparison. Absorbance values were used in the range of (0.08) to (0.10) which were associated with (10^7) to (10^8) number of bacteria per mL of MHA. The sterile discs approximately (15) mm in diameter was then placed over the swabbed MHA plates. Incubation occurred at (37)˚C for 24 hour so as to grow the bacteria. Antibacterial activity has been recorded through the measurement of the inhibition zone diameter (mm). This work done in Al-Ameen Center for Researches and Advanced Biotechnologies/Najaf Governorate - Iraq country.
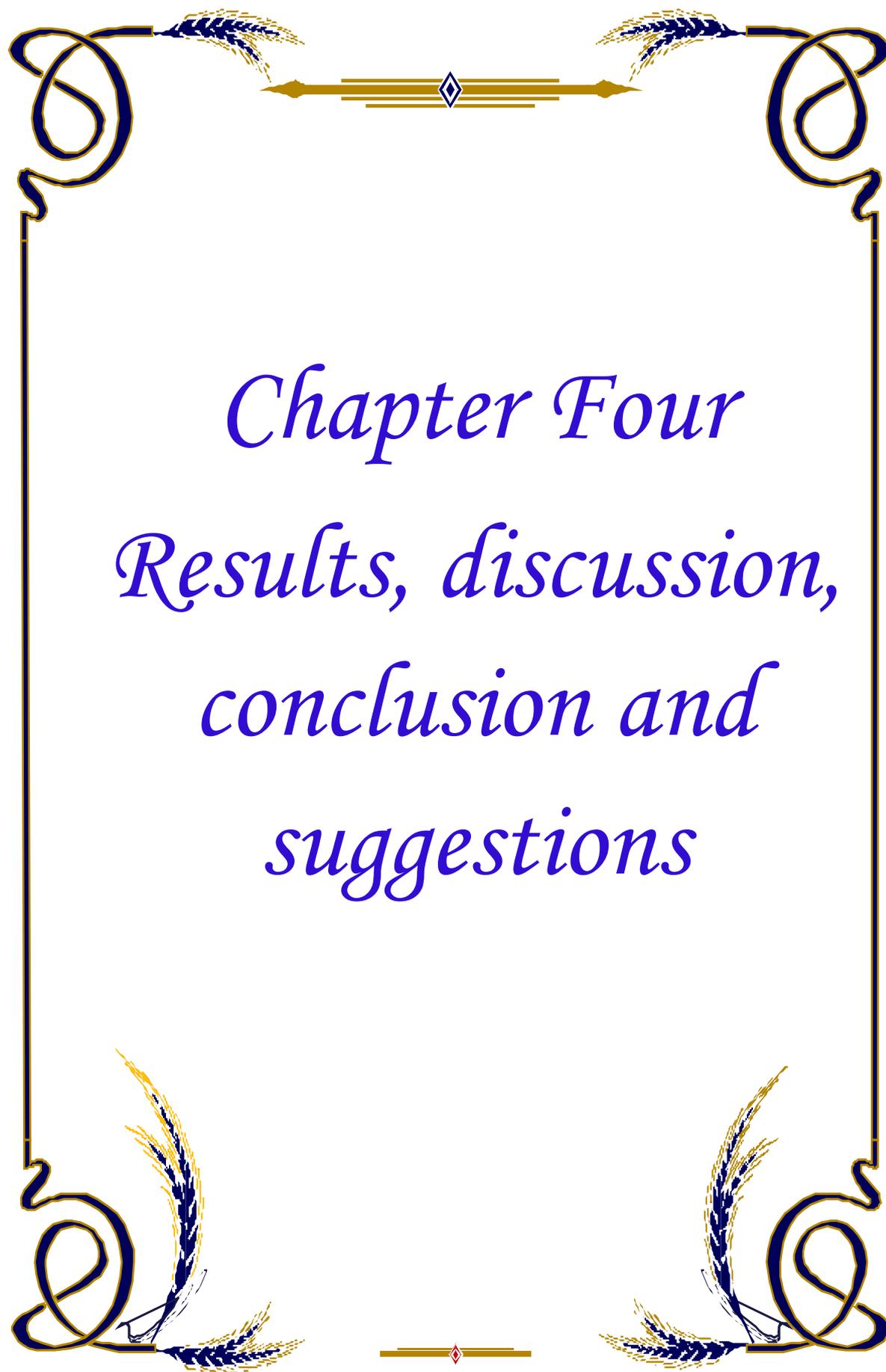
Chapter Four

Results, discussion, conclusion and suggestions

4.1Introduction

This chapter involves the results of structural properties, D.C electrical and optical measurements for (PANI/ okra plant waste) blend and (PANI/okra plant waste /Ag) nanocomposites and discussions. Beside that discuss each of the medical application and antibacterial activity.

4.2 The Structural Properties

4.2.1 Scanning Electron Microscope (SEM)

The morphology of (PANI/ okra plant waste) blend and (PANI/okra plant waste /Ag) nanocomposites was observed by scanning electron microscopy (SEM) are shown in Figure (4.1). The SEM micrograph of okra plant Figure (4.1A) clearly demonstrated the presence of oriented unit cells with more or less parallel orientations and show well-organized networks where the intercellular space is filled up by the okra plant fiber the structure of a okra plant consists of several elementary fibers overlapped along the length of the fibres and bonded together, by pectin and other compounds that give strength to the bundle as a whole and increased their surface area surface roughness and in addition, porosity on the fiber surface [39,113].

Figure (4.1B) shows that the surface of the films is not smooth and showing distinct structures pointed by arrows, these structures refer to irregular morphology [114,115]. Figure (4.1C) shows the SEM image of the polymer blend has pores. These pores represent the crystalline region in the blend. Further, the uniform distribution of these pores over the total surface of the blend indicates that the (PANI/ okra plant waste) blends forms a homogeneous structure due to polymer-plant waste interactions which confirm that the okra plant is miscible with PANI, that led showed a rough surface [116,117]. Figures (4.1D, E, F and G) display addition (0.5%, 1%, 1.5% and 2%) of Ag nanoparticles to the (PANI/okra plant waste) blend.

From the SEM image was observed exhibit varying microstructures contains grains   consisted of particles with different sizes and clustered   [115,118]. these results are disagreement with [56].
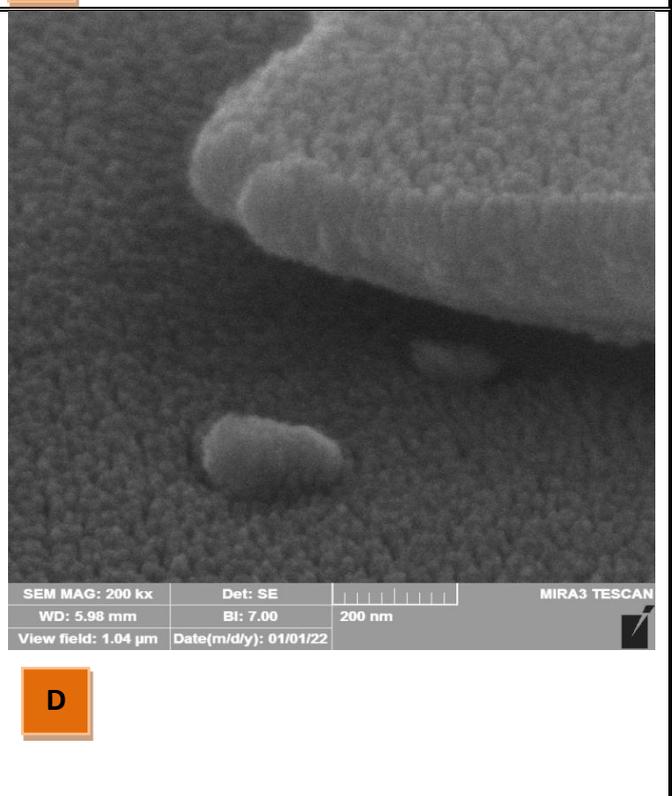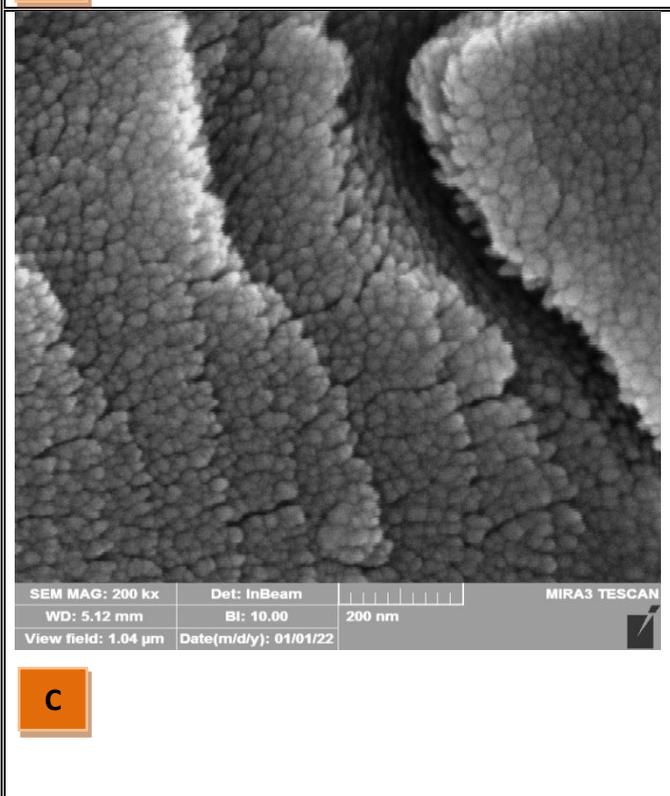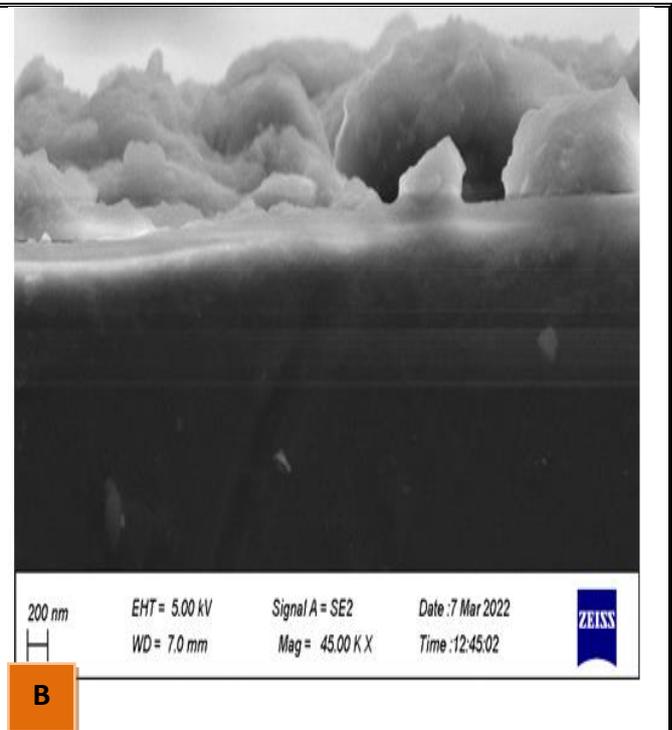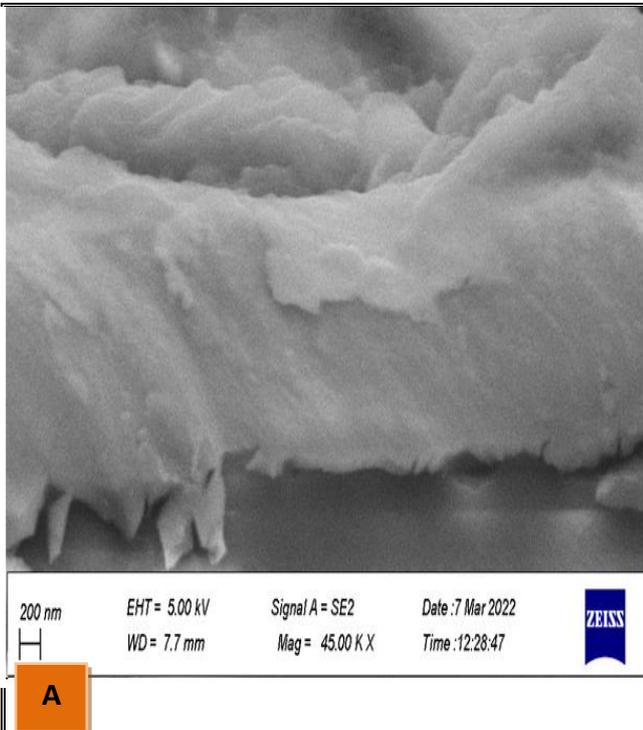
Figure (4.1): SEM images of (A) Okra plant (B) PANI (C) (PANI/ okra plant waste) blend (D) (PANI/okra plant waste/0.5%Ag) nanocomposite (E) (PANI/ okra plant waste /1%Ag) nanocomposite (F) (PANI/ okra plant waste /1.5%Ag) nanocomposite (G) (PANI/ okra plant waste /2%Ag) nanocomposite.

4.2.2 Atomic Force Microscope (AFM)

Figures (4.2) and (4.3) shows a 2D and 3D micrograph respectively of the (PANI/ plants waste) blends and (PANI/ plants waste /Ag) nanocomposites films with different ratio of Ag which showed a uniform granular surface morphology. The roughness values (root mean square RMS), mean height and maximum height of the samples are listed in table (4.1). It may be noted that this value of the roughness is increasing with okra plant which indicates increased available surface of the (PANI/ plants waste) blends. The high value of roughness observed indicates porous morphology of the film [119].
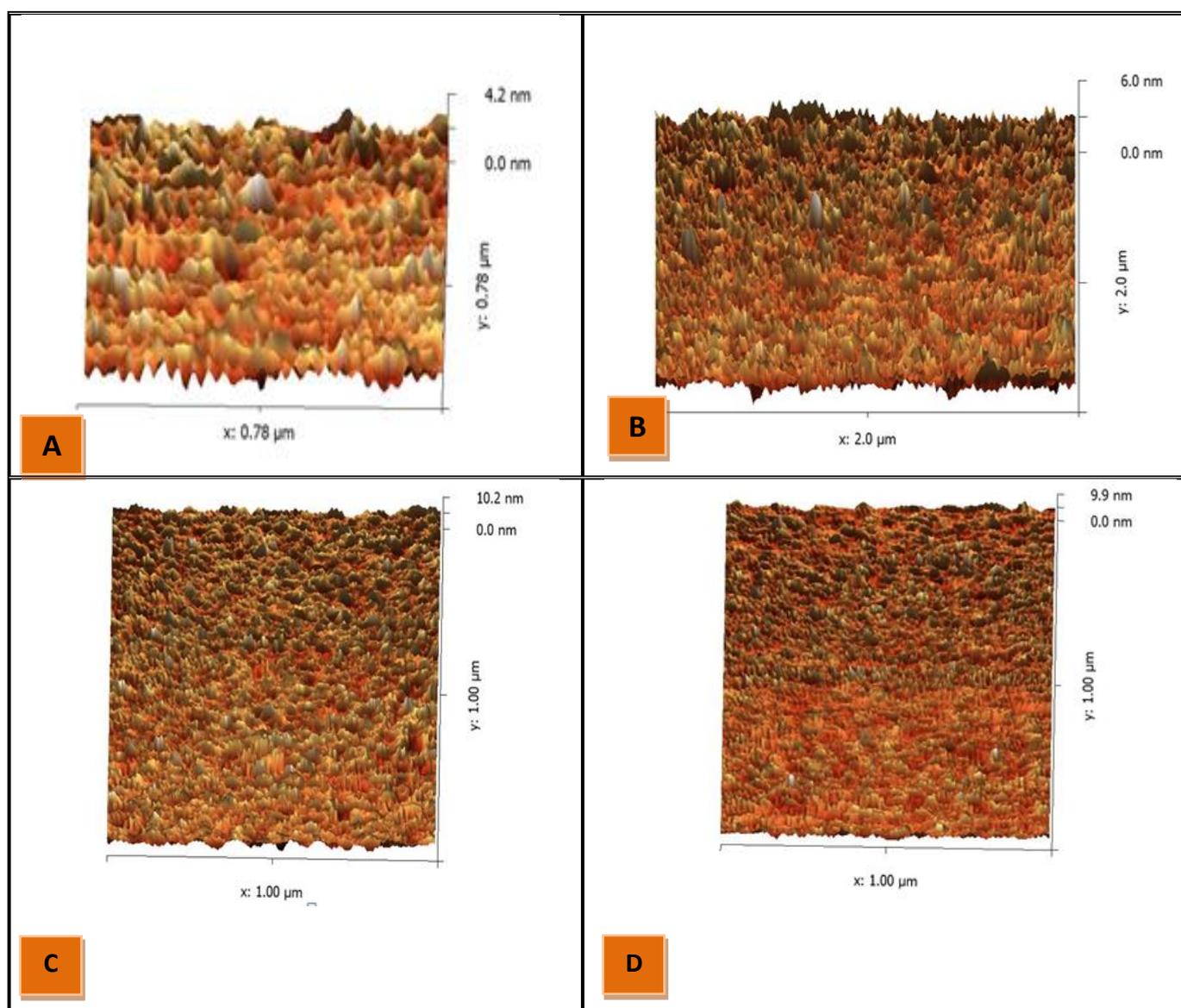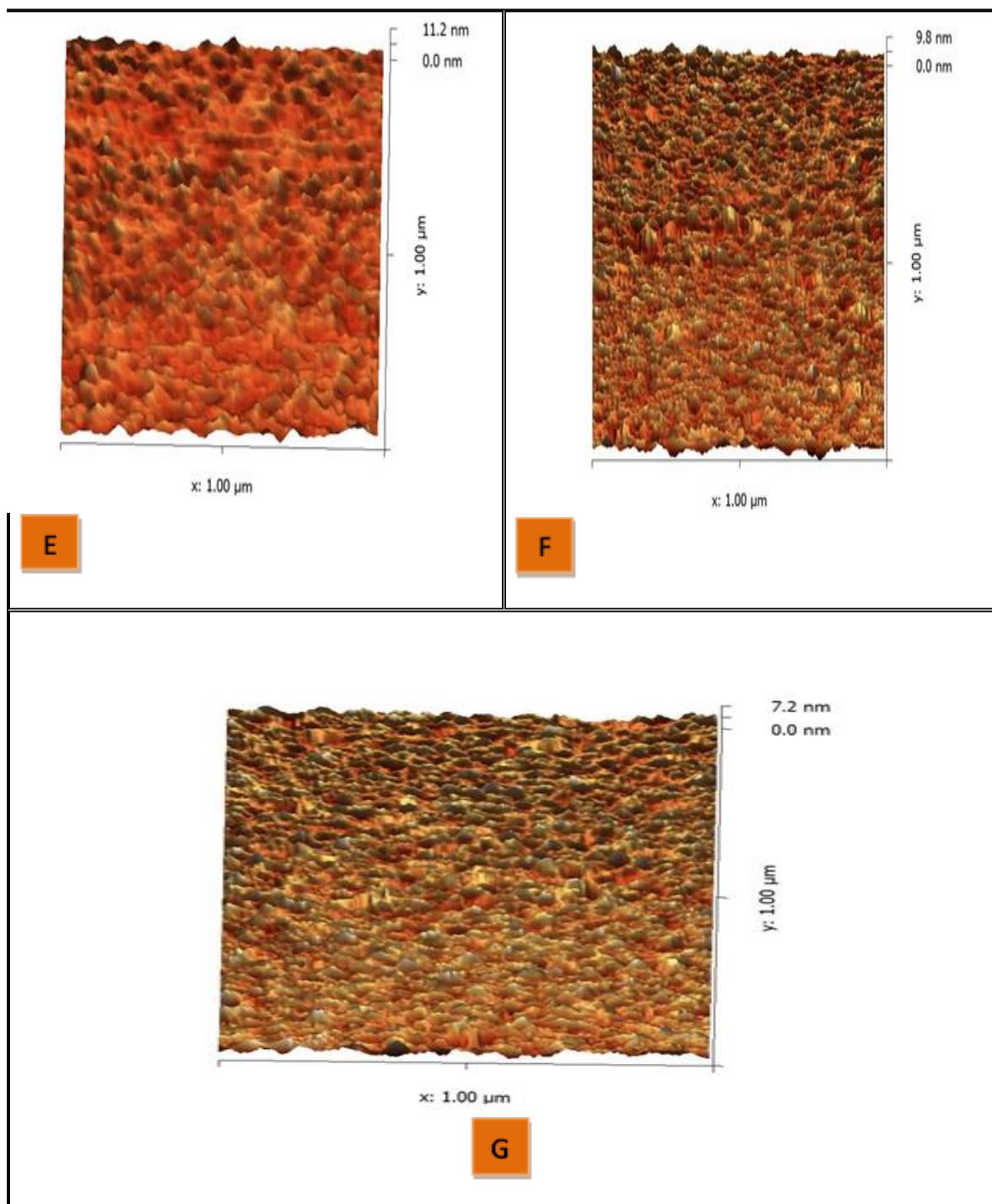
Figure (4.2): 2-D images for of (A) Okra plant (B) PANI (C) (PANI/ okra plant waste) blend (D) (PANI/okra plant waste/0.5%Ag) nanocomposite (E) (PANI/ okra plant waste /1%Ag) nanocomposite (F) (PANI/okra plant waste/1.5%Ag) nanocomposite (G) (PANI/ okra plant waste /2%Ag) nanocomposite.
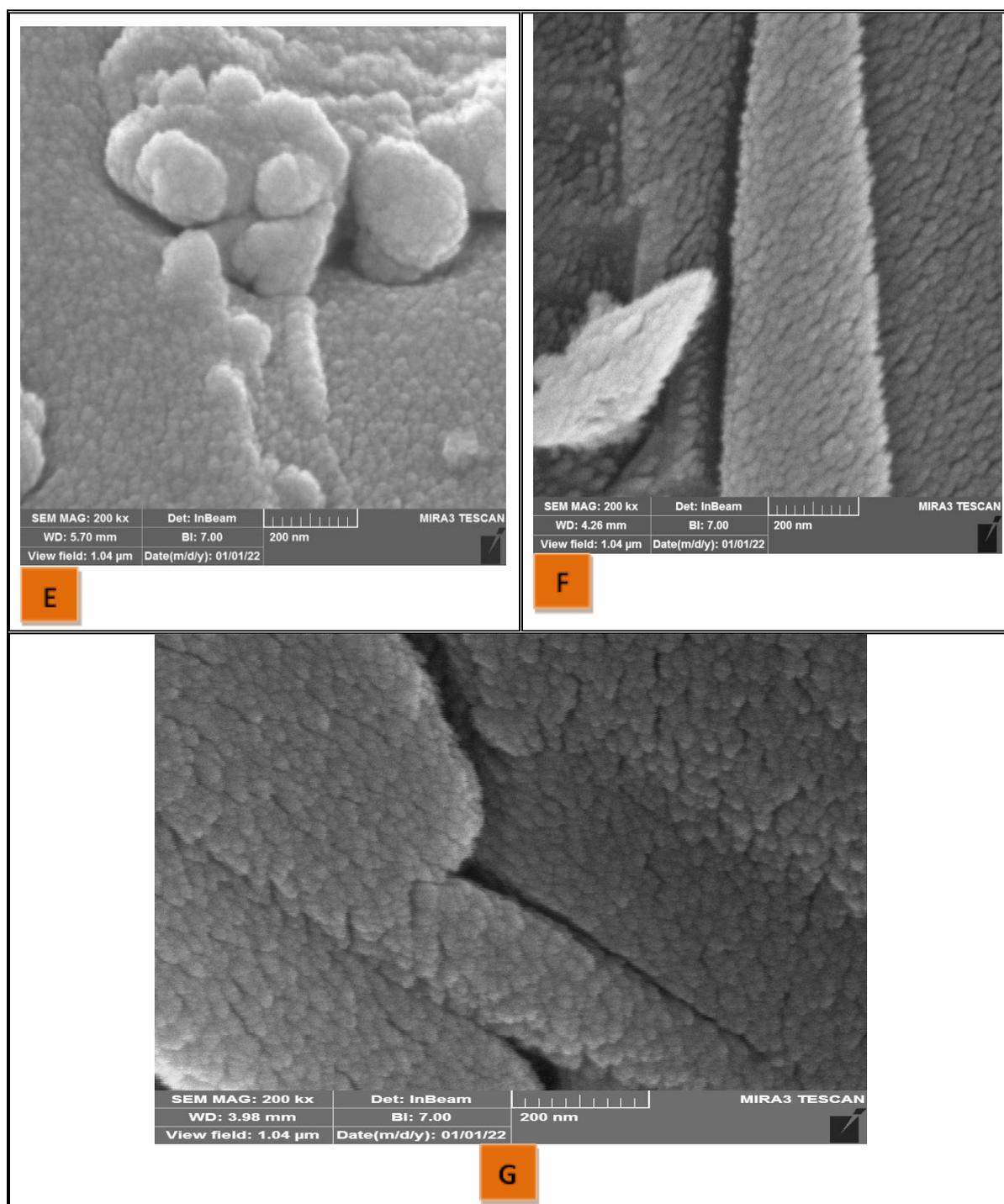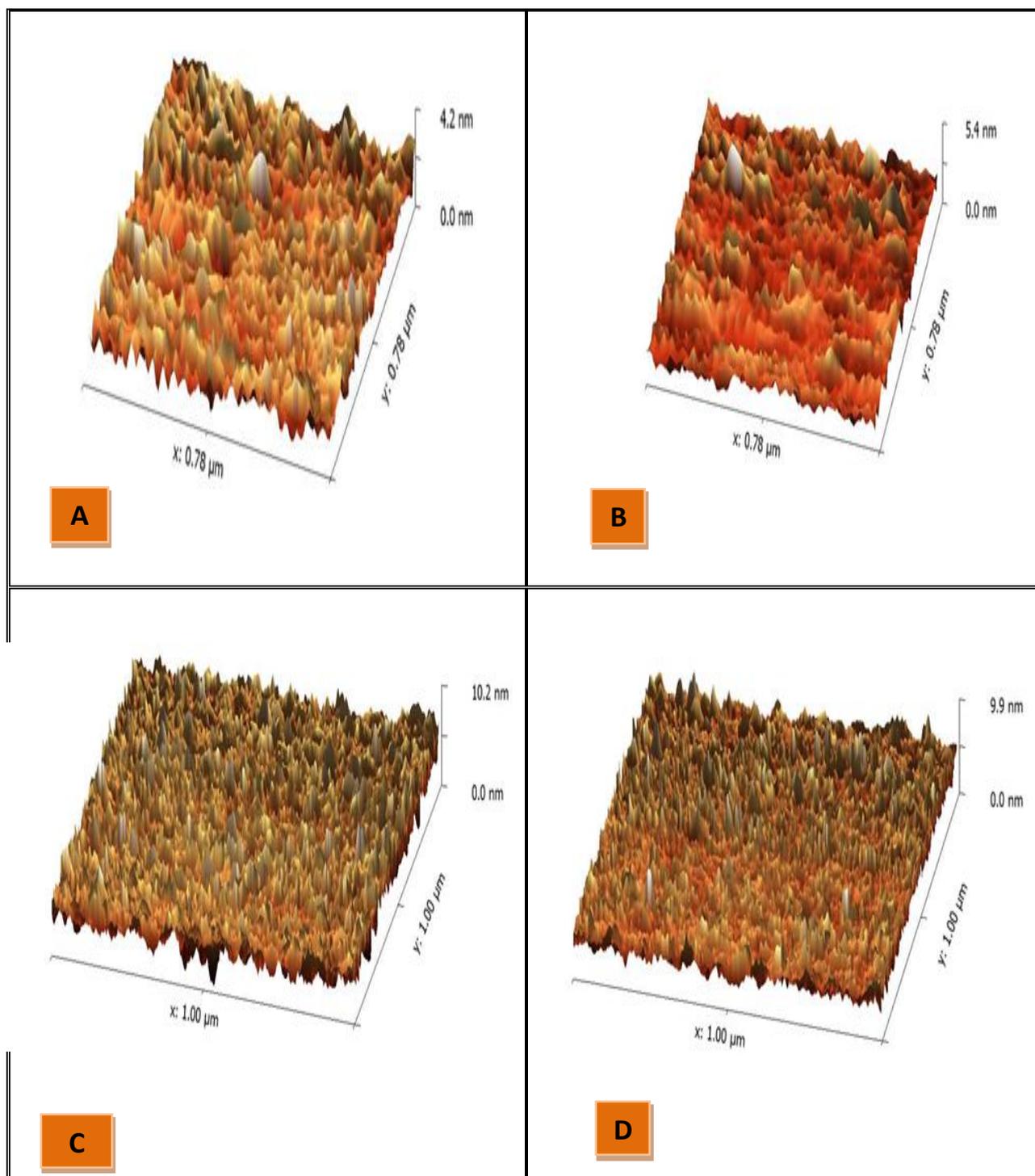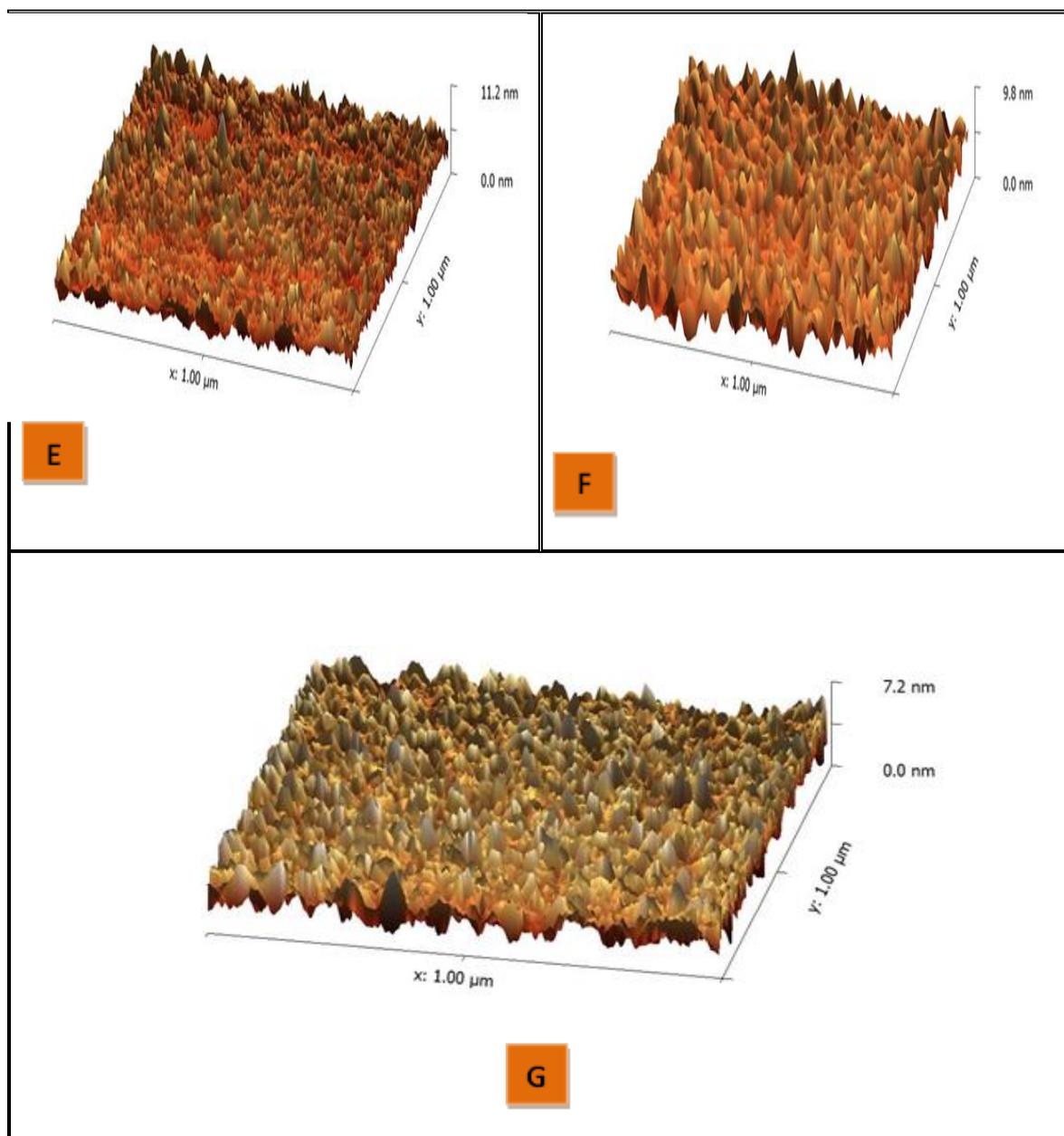
Figure (4.3): 3-D images of (A) Okra plant (B) PANI (C) (PANI/ okra plant waste) blend (D) (PANI/okra plant waste/0.5%Ag) nanocomposite (E) (PANI/okra plant waste /1%Ag) nanocomposite (F) (PANI/ okra plant waste/1.5%Ag) nanocomposite (G) (PANI/okra plant waste/2%Ag) nanocomposite.

Table (4.1): AFM data for (PANI/okra plant waste) blend and (PANI/okra plant waste /Ag) nanocomposites thin films.

Sample	Mean Height (Sa) (nm)	Root Mean Square (RMS) (nm)	Maximum Height (Sz) (nm)
Okra plant	8.51	14.24	109.6
PANI	1.67	2.01	12.02
(PANI/okra plant waste) blends	5.55	6.75	24.77
(PANI/okra plant waste /0.5%Ag) nanocomposites	1.34	1.71	14.05
(PANI/okra plant waste /1%Ag) nanocomposites	3.35	4.38	28.92
(PANI/okra plant waste /1.5%Ag) nanocomposites	3.43	4.28	27.33
(PANI/okra plant waste /2 %Ag) nanocomposites	4	4.76	24.77

4.3 The Optical Properties

The optical properties of (PANI/okra plant waste) blend (PANI/okra plant waste/Ag) nanocomposites include absorbance, transmittance, absorption coefficient, energy gap, extinction coefficient, refractive index, dielectric constants and optical conductivity.

4.3.1 Absorbance Spectrum (A) and Absorption Coefficient(α)

Figure (4.4 A and B) and (4.5 A and B) display the variation of absorbance and absorption coefficient for (PANI/okra plant waste) blend and (PANI/ okra plant waste/Ag) nanocomposites with wavelength (λ) of the incident light respectively. The absorbance values and absorption coefficient are high in the (PANI/okra plant waste) blend, while they are lower in the (PANI/ okra plant waste / Ag) nanocomposites. The findings show that the okra plant as a filler for polymer matrices and contains a variety of organic reducing agents in various combinations and concentrations [120,121]. In this system, initiates polymerization to produce a new polymeric material [122]. where the chemical groups lignocellulose fiber components of the okra plant appear absorption bands [39,120]. This finding suggests that the okra plant may interact with polyaniline units, resulting appear absorption bands.
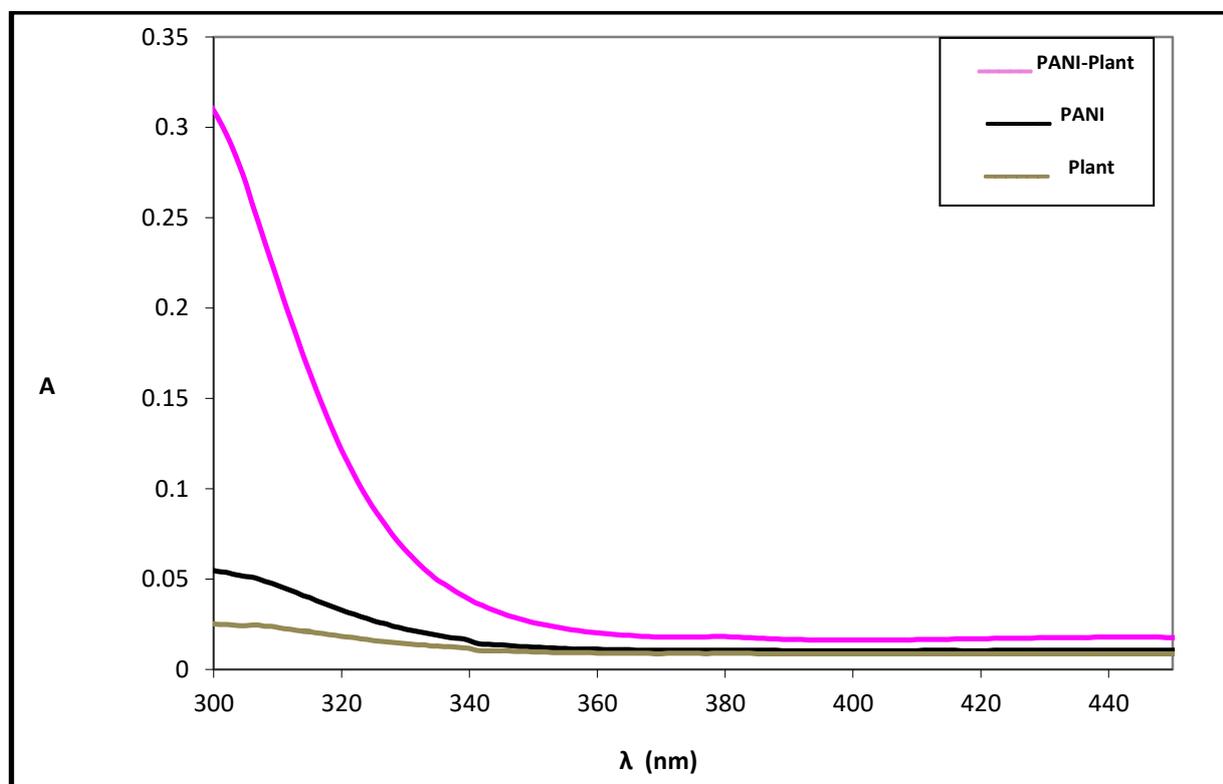
Figure (4.4 A): Absorbance spectrum as a function of the wavelength (PANI /okra plant waste) blend
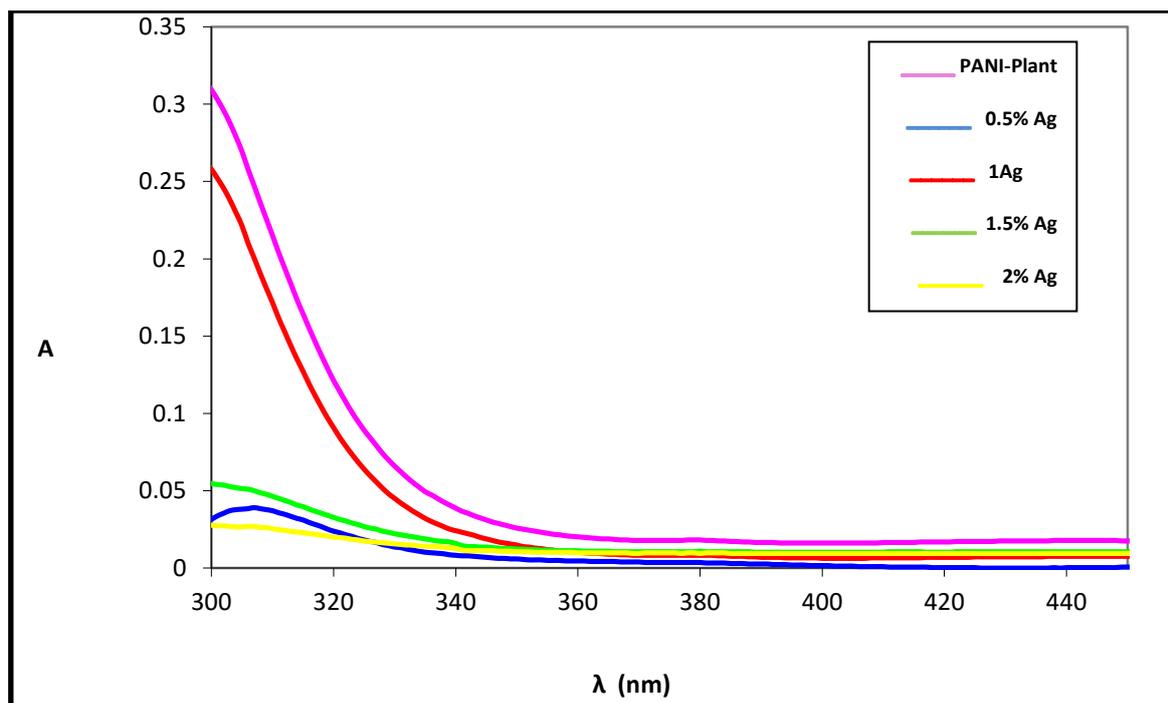
Figure (4.4 B): Absorbance spectrum as a function of the (PANI/okra plant waste /Ag) nanocomposites.
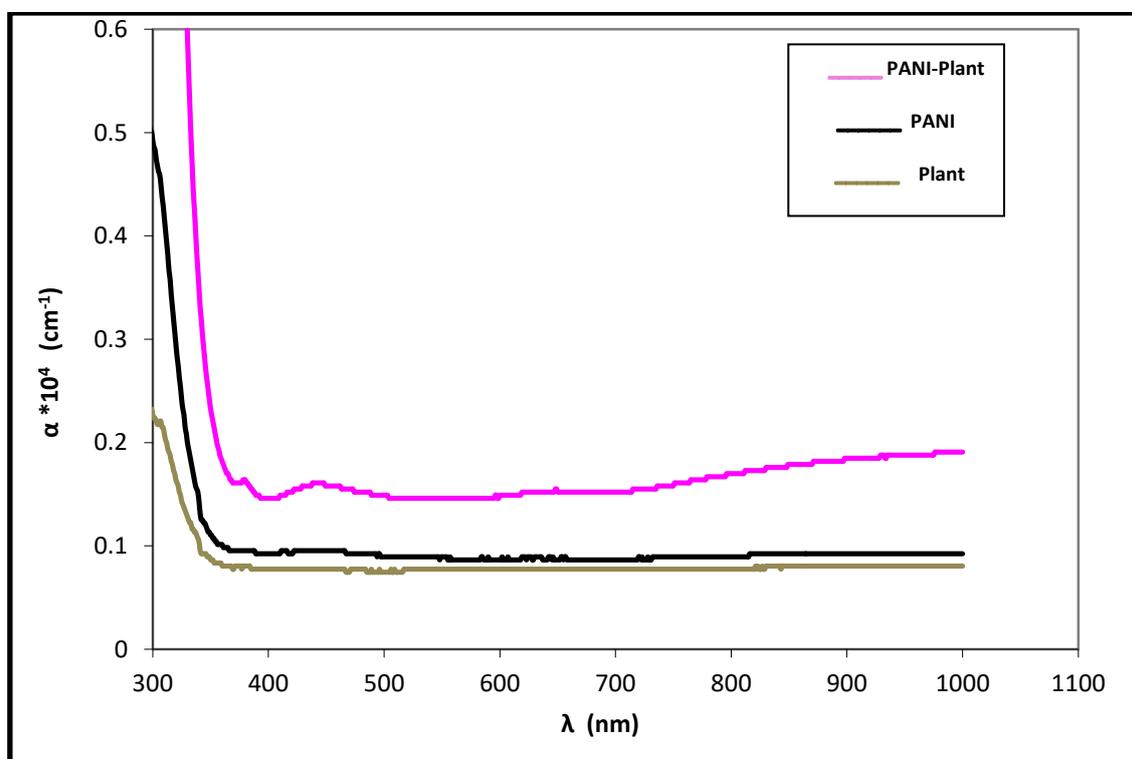
Figure (4.5A): Absorption coefficient spectrum as a function of the wavelength (PANI / okra plant waste) blend
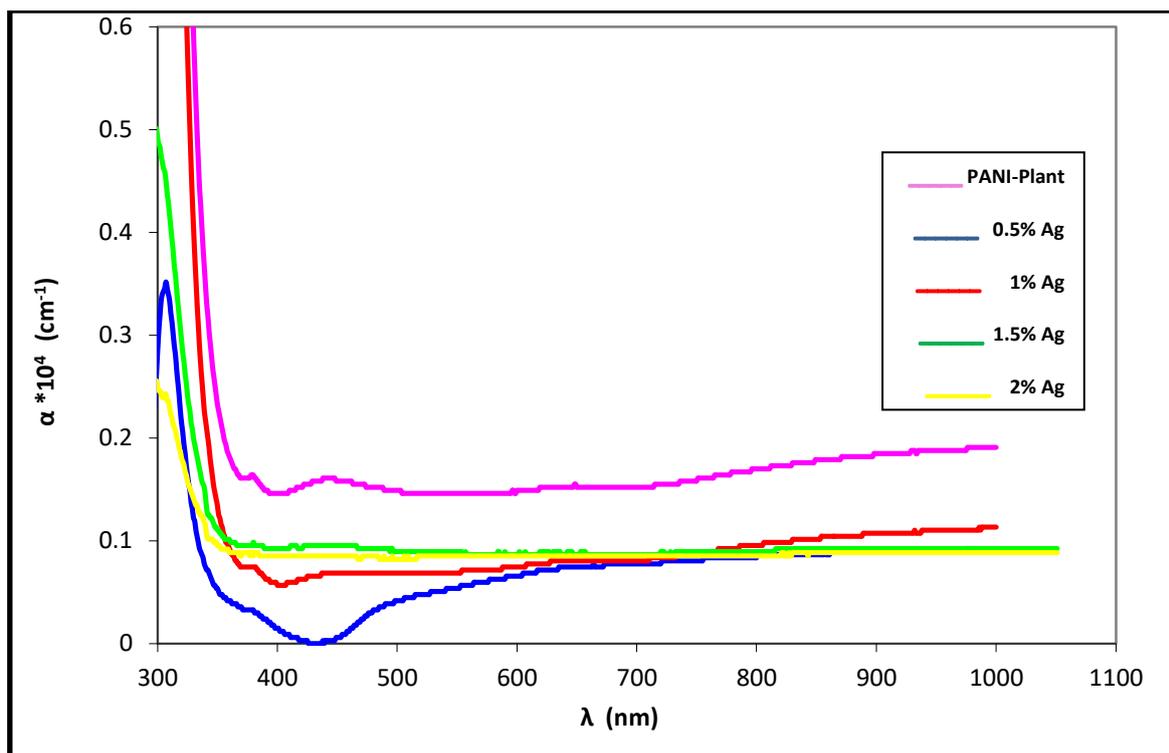
(B)

Figure (4.5B): Absorption coefficient spectrum as a function of the wavelength (PANI/okra plant waste /Ag) nanocomposites.

4.3.2 Transmittance Spectrum (T)

Figure (4.6 A and B) display the variation of transmittance (T) for (PANI/ okra plant waste) blend and (PANI/okra plant waste / Ag) nanocomposites with the wavelength (λ) of the incident light. As shown in the figure below the transmittance spectrum exhibits an opposite behavior of absorbance behavior, where the transmittance decreases with (PANI/ okra plant waste) blend also the value of transmittance increasing with the increase of the wavelengths, as a result of electronic transitions, it is observed that transmittance decreases with higher wavelengths [123].
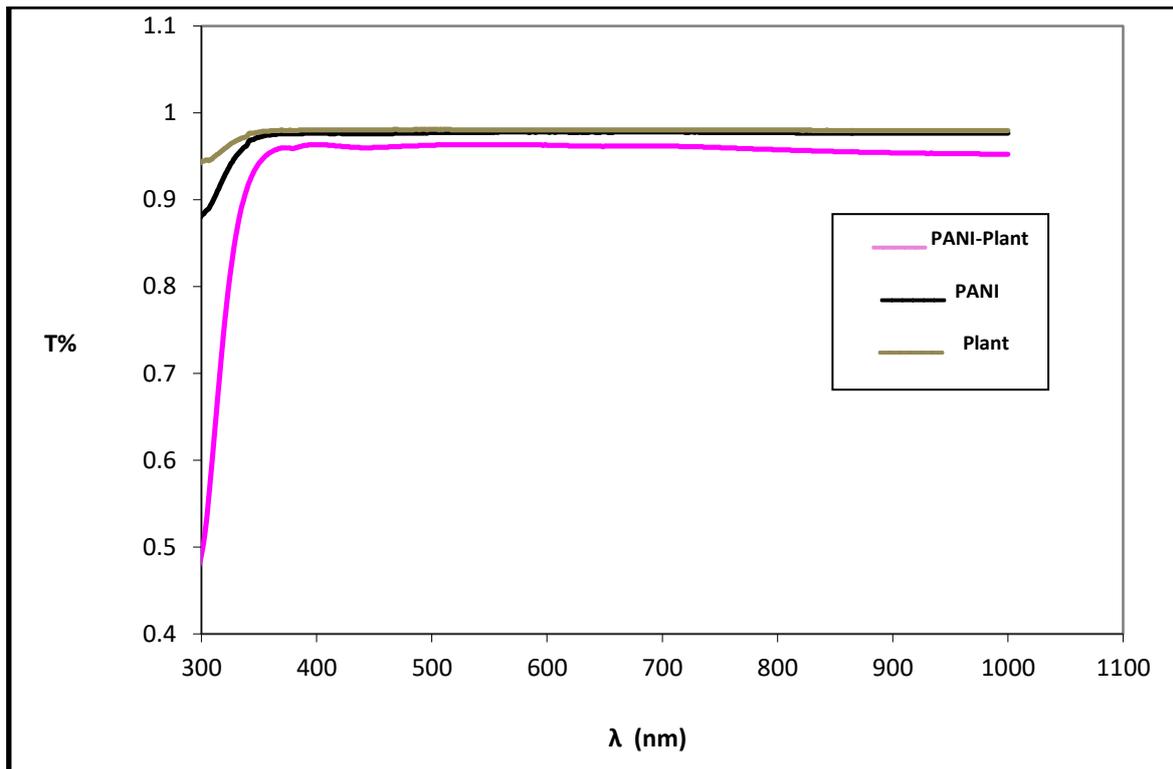
Figure (4.6A): Shows the transmittance spectra (PANI/okra plant waste) blend
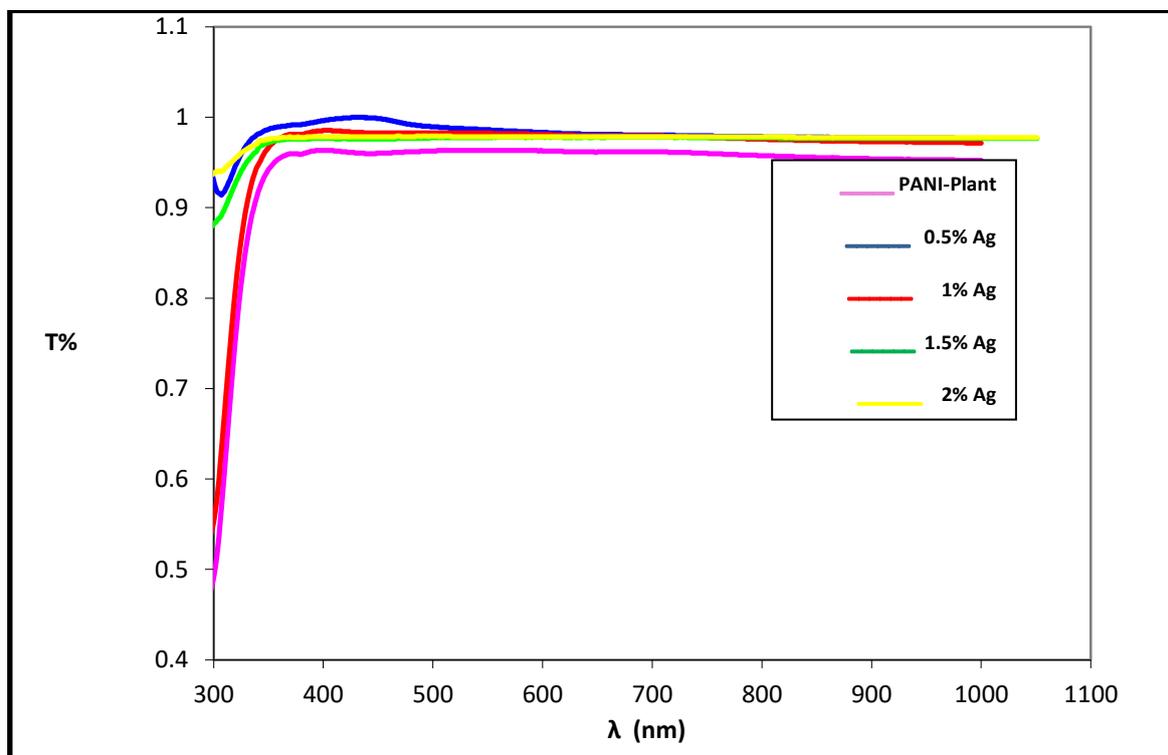
Figure (4.6B): Shows the transmittance (PANI/okra plant waste/Ag) nanocomposites.

4.3.3 Energy Gap

The values of the optical energy gap of thin films are measured by using the equation (2-17). For all prepared thin films, optical energy gap is determined by drawing between $(\alpha h\nu)^2$ as a function of photon energy, by stretching straight curve line and an intersection with x-axis which gives the energy gap value for all prepared thin films. The energy gaps for allowed indirect transitions of (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites are shown in figure (4.7A and B). The values have been getting in table (4.2) it's showed that the values of allowed direct energy gap of (PANI/okra plant waste) blend decrease and it is increase with (PANI/okra plant waste/Ag) nanocomposites, this decrease can be explained by the formation of localized levels in the energy gap [124]. In this case, the transition of electrons takes place in two stages involving the transition from the valence band to the local levels in the energy gap and the other transition from the local levels to the conduction band as a result of the that okra plant enhances the conductivity in the (PANI/okra plant waste) blend.
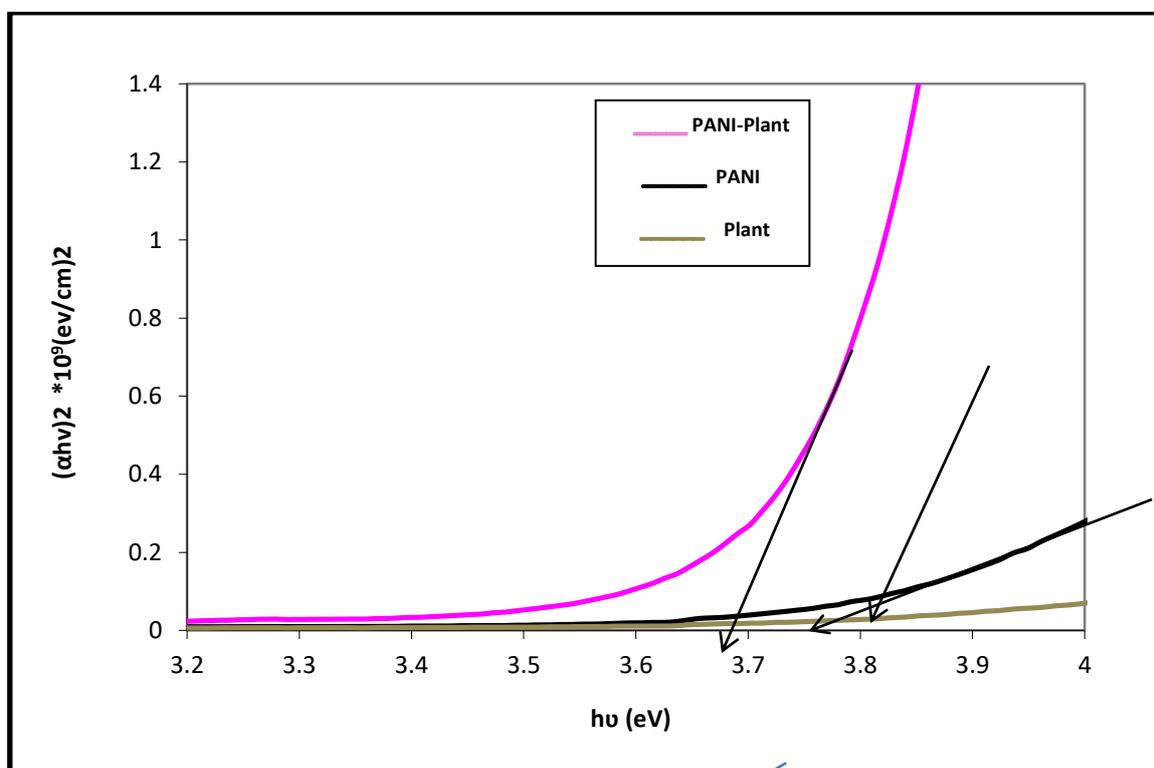
Figure (4.7A): Allowed direct energy gap of (PANI/okra plant waste) blend
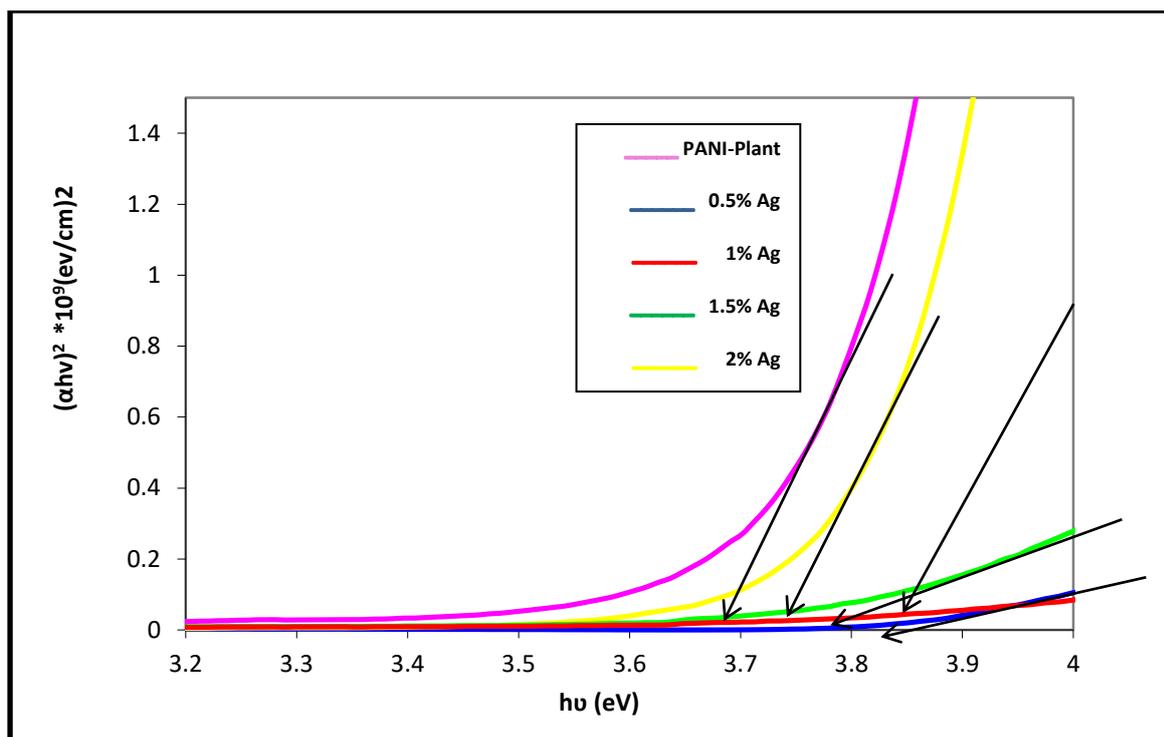
Figure (4.7B): Allowed direct energy gap of (PANI/okra plant waste/Ag) nanocomposites.

Table (4.2): The allowed direct energy gap of (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites.

Samples	Energy Gap(ev)
(PANI / okra plant waste) blend	3.690
PANI	3.815
Okra plant	3.780
(PANI/ okra plant waste/0.5%Ag) nanocomposite	3.887
(PANI/ okra plant waste/1%Ag) nanocomposite	3.862
(PANI /okra plant waste /1.5% Ag) nanocomposite.	3.803
(PANI/okra plant waste/ 2%Ag) nanocomposite.	3.746

4.3.4 Refractive Index (n)

The refractive index is measured using the equation (2-8). The refractive index of (PANI/okra plants waste) blend and (PANI/okra plant waste/Ag) nanocomposites as a function of wavelength is shown in figure (4.8 A and B).

The figure display that the refractive index increase with (PANI/okra plant waste) blend, this increase can be attributed on the basis of okra plant which in turn increase the degree of cross-linking between chains polymer which increases the speed of light propagation through it [121,123].
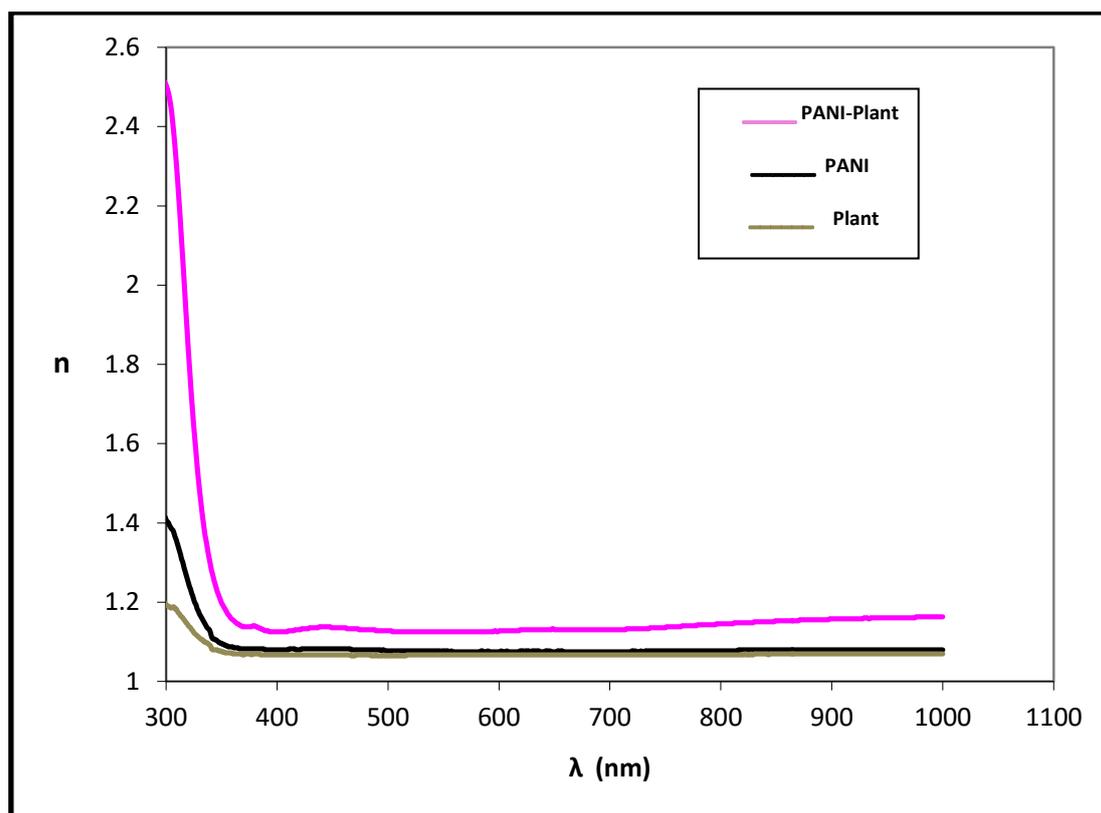
Figure (4.8A): Illustrates refractive index (PANI /okra plant waste) blend.
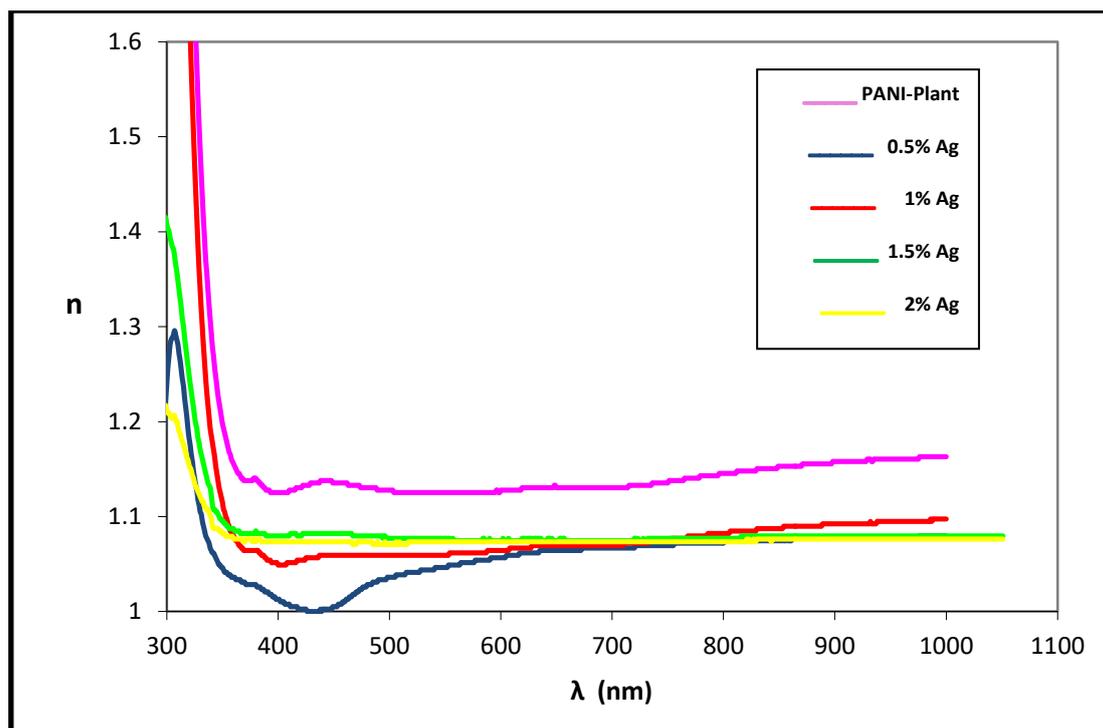
Figure (4.8B): Illustrates refractive index (PANI/okra plant waste/Ag) nanocomposites.

4.3.5 Extinction Coefficient (k)

The extinction coefficient (k) is calculated using the equation (2-7). figure (4.9 A and B) display the extinction coefficient for (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites as a function of wavelength (λ) respectively. The figure shows that increase of the extinction coefficient of (PANI/ okra plant waste) blend, due to high absorption coefficient for (PANI/okra plant waste) blend. Where okra plant will modify the structure of the host (PANI/okra plant waste) blend [120].
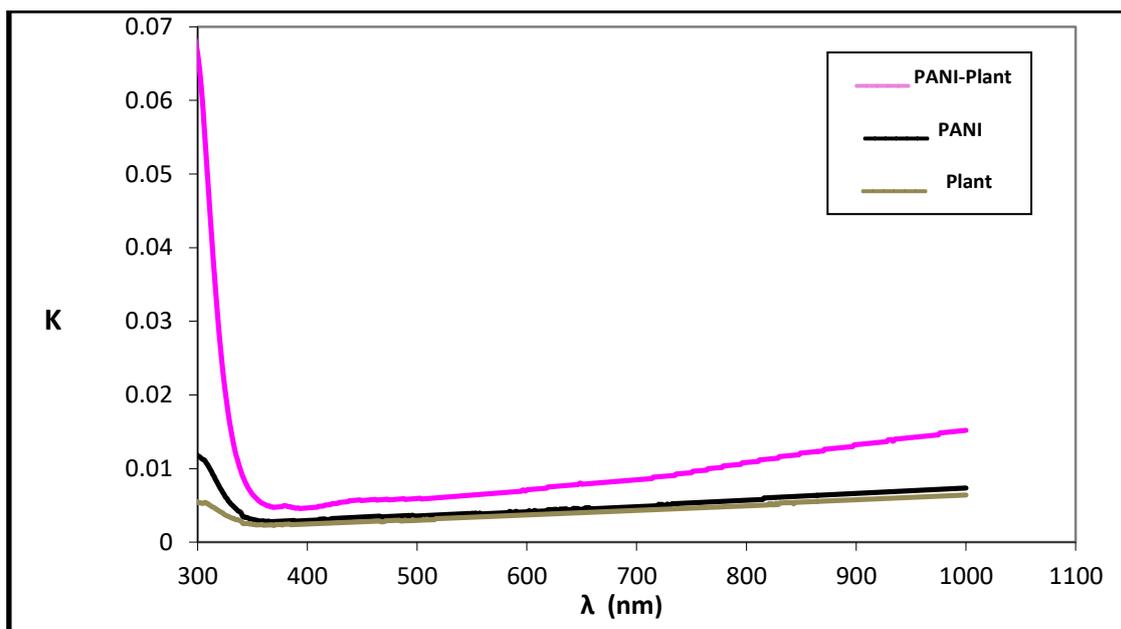
Figure (4.9A): Extinction coefficient variation with wavelength of (PANI/okra plant waste) blend.
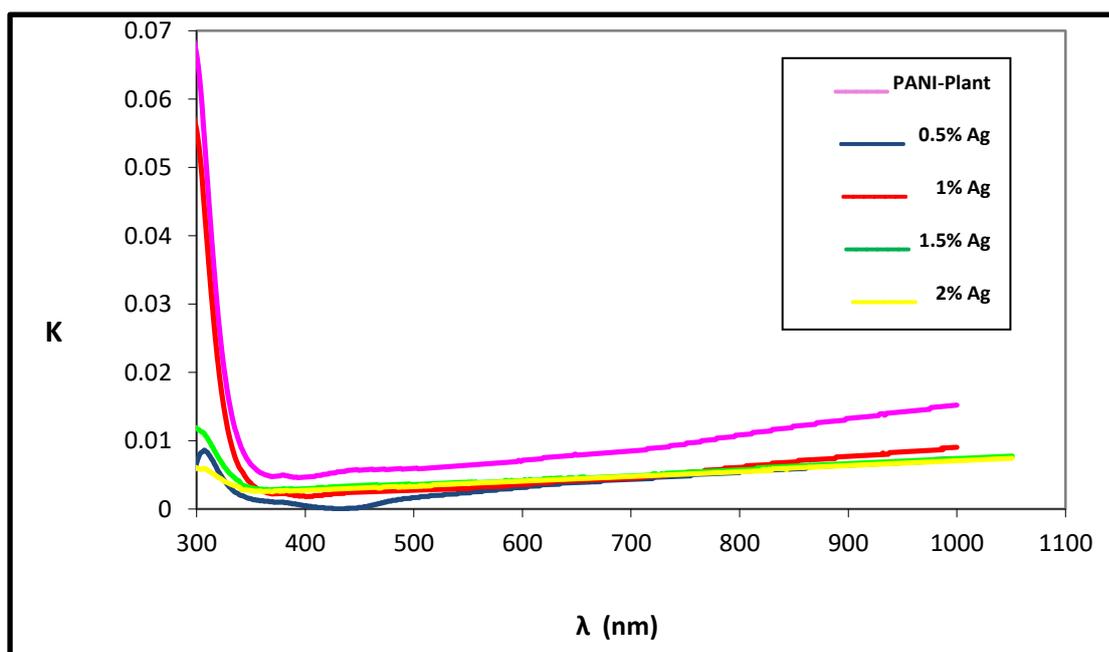
Figure (4.9B): Extinction coefficient variation with wavelength of (PANI/okra plant waste/Ag) nanocomposites.

4.3.6 Real and Imaginary Parts of Dielectric constant

Figures (4.10 Aand B) and (4.11 A and B) shows the variations of real and imaginary part ($\mathcal{E}_r$, $\mathcal{E}_i$) respectively with wavelength for (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites. The real and imaginary

parts of dielectric constant are measured by using equations (2-13) and (2-14) respectively. The behavior of the real part of the dielectric constant is similar to that of the refractive index (n) because the value of the extinction coefficient (k^2) is very small compared with refractive index (n^2), while the imaginary part of the dielectric constant is essentially proportional with extinction coefficient (k) values [125]. From the Figures below are noted that real and imaginary parts increase with (PANI/okra plant waste) blend this is due to increase the absorption coefficient of (PANI/okra plant waste) blend [92] and is noted that real and imaginary parts decrease with nanoparticles which indicates that the samples have no same structure. Hence, the change in the additive of nanoparticles gave change in the chemical composition of the (PANI/okra plant waste/Ag) nanocomposites [126].
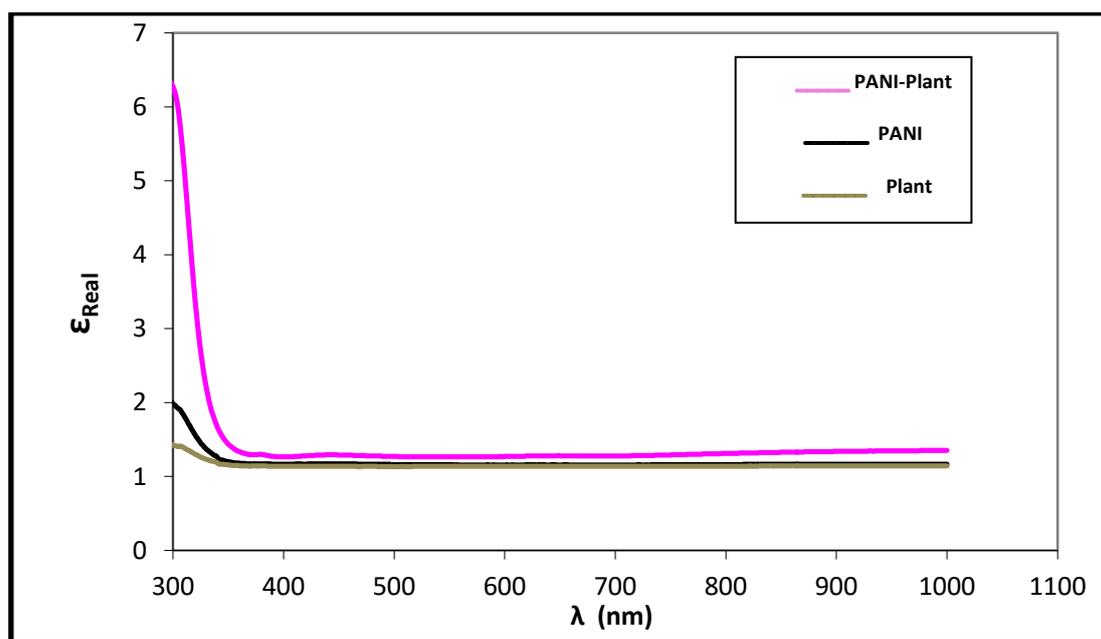
Figure (4.10 A): Real part of dielectric constant variation of (PANI/okra plant waste) blend.
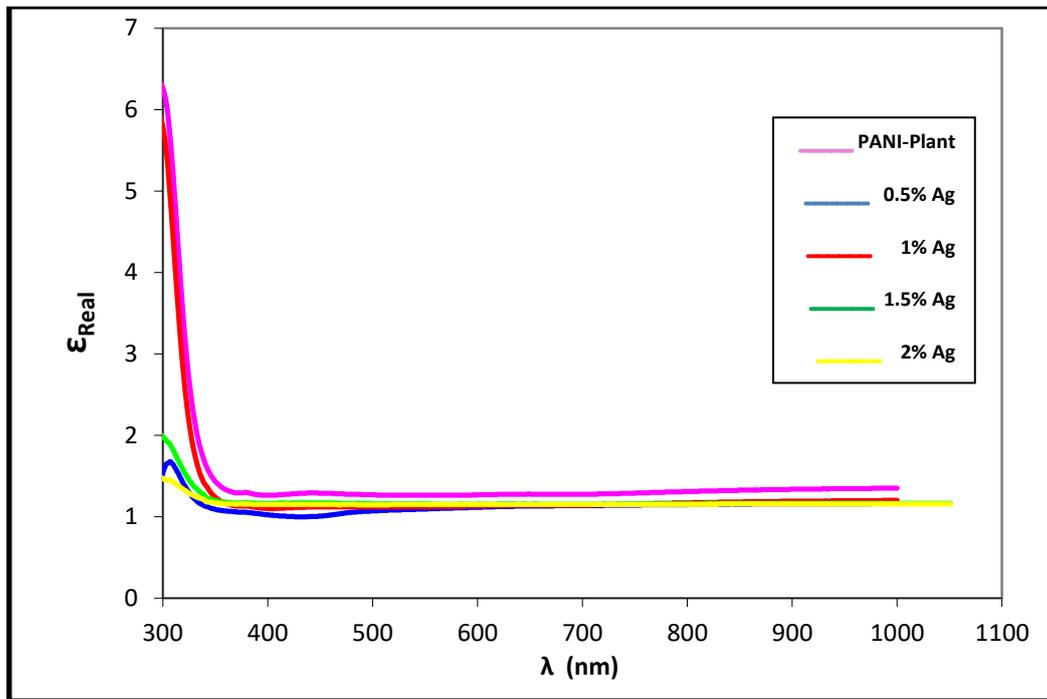
Figure (4.10 B): Real part of dielectric constant variation of (PANI/okra plant waste/ Ag) nanocomposites with wavelengths.
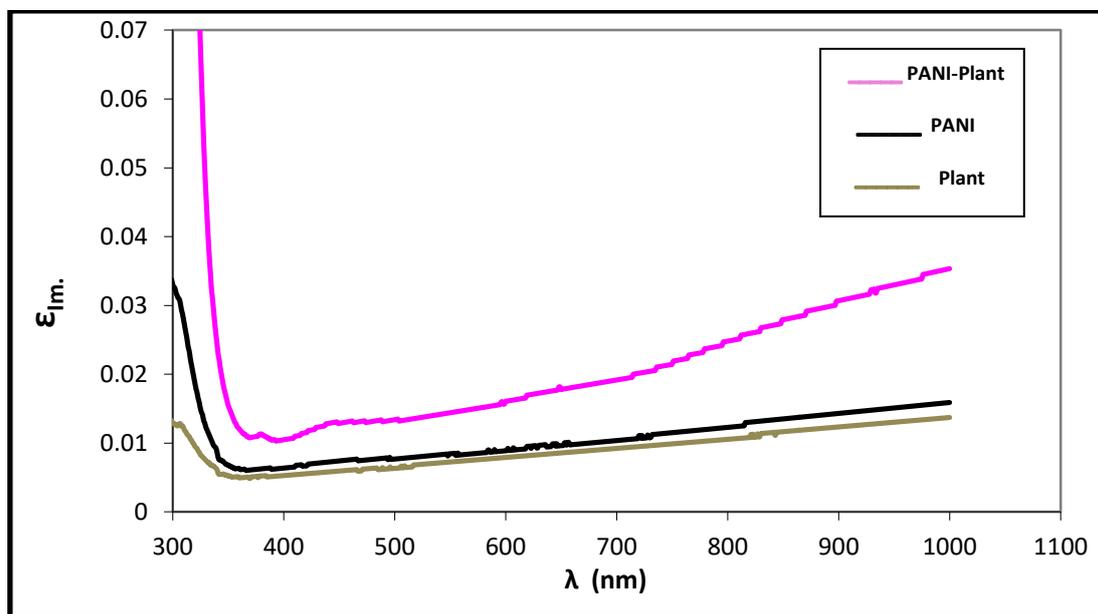
Figure (4.11A): Imaginary part of dielectric constant variation for (PANI/okra plant waste) blend.
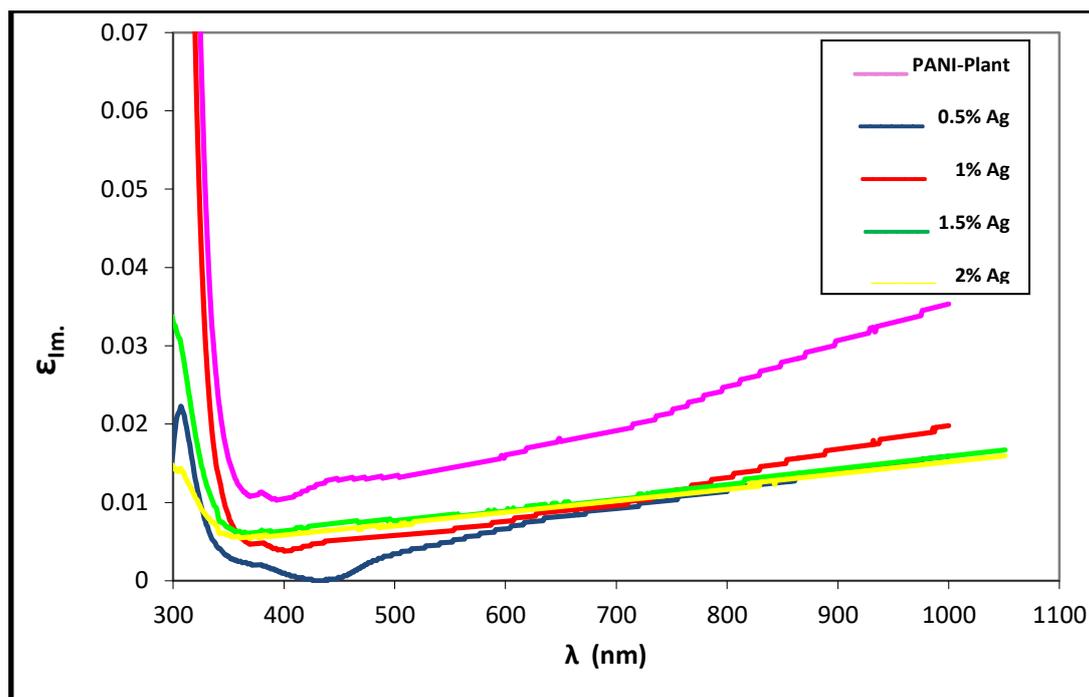
Figure (4.11B): Imaginary part of dielectric constant variation for (PANI/okra plant waste/Ag) nanocomposites with wavelengths.

4.3.7 Optical Conductivity

The optical conductivity of a material can be calculated by using the equation (2-16). Figure (4.12 A and B) display the variation of optical conductivity with the wavelength for (PANI/ okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites. The figure show that the optical conductivity of (PANI/okra plant waste) blend is increased, this behavior attributed to the formation of localized levels in the energy gap; that causes increase in the density of localized levels in the energy gap, thus, increase of the absorption coefficient consequently increasing the optical conductivity [127]. The optical parameters increase while transmittance and energy gap decrease of (PANI/okra plant waste) blend with additive okra plant to polymer compared with nanocomposites, these results are disagreement with [56].
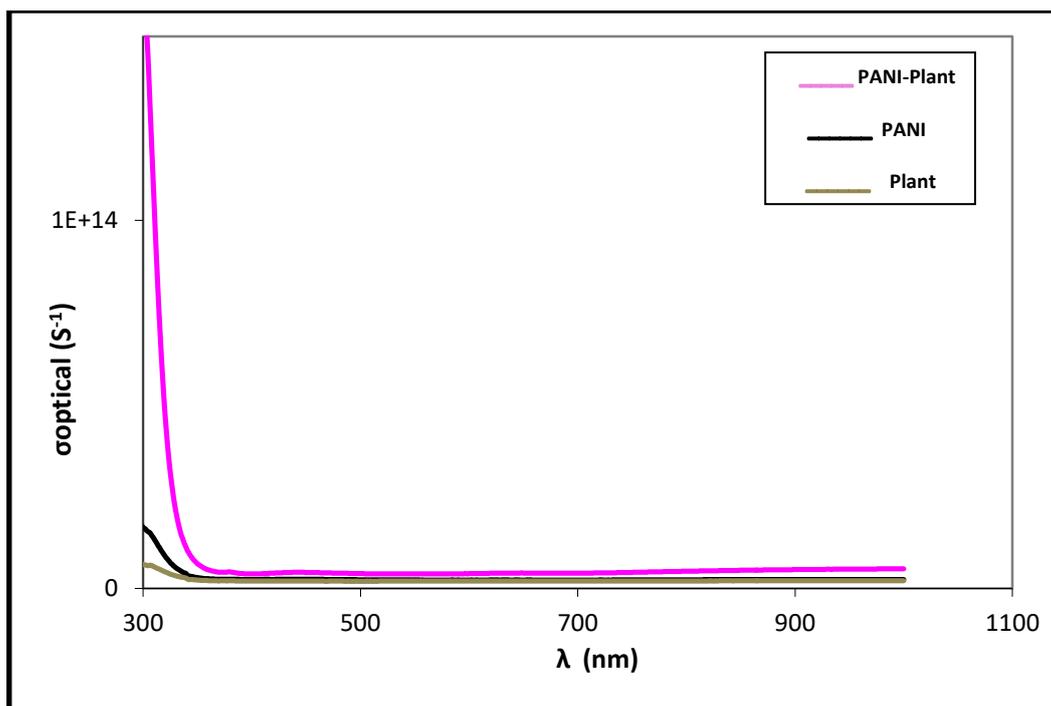
Figure (4.12A): Optical conductivity as a function of wavelength for (PANI/plants waste) blend.
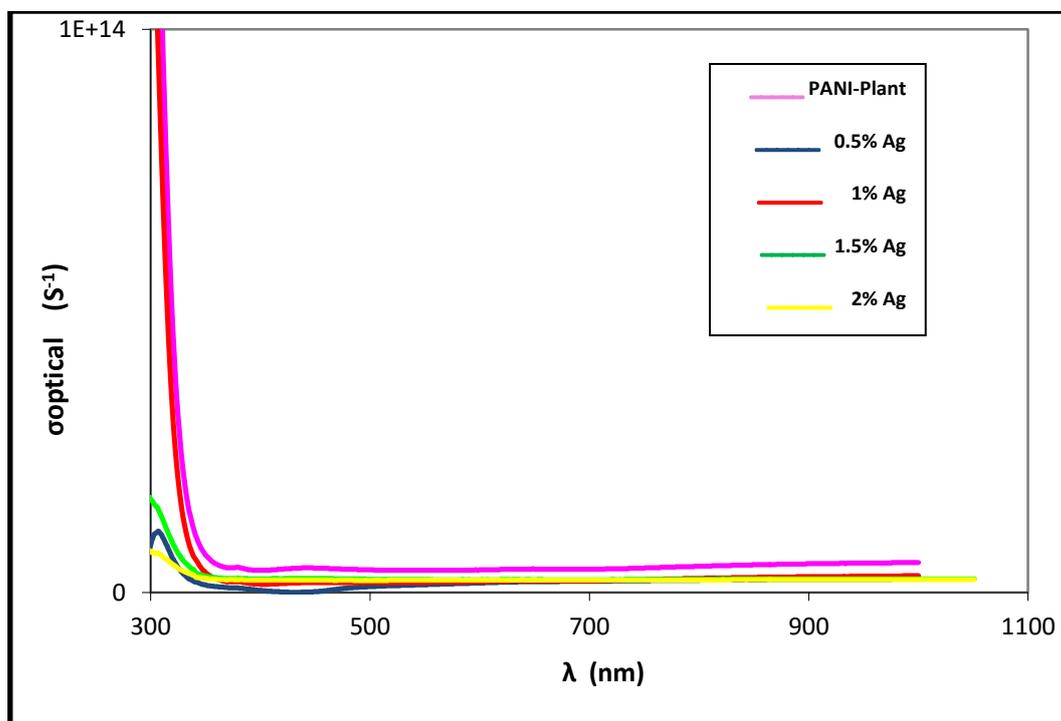
Figure (4.12B): Optical conductivity as a function of wavelength for (PANI/plants waste /Ag) nanocomposites.

4.4 I-V Characterization

Figure (4.13) shows I-V characteristics taken by using thin films method on the prepared film of the (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites. Figure (4.13AandC) shows the I-V characteristics of okra plant waste and (PANI/okra plant waste) blends which clearly indicates that ohmic contact has been established the PANI and okra plant waste. Electron transfer, which readily occurs on the surface of okra plants fibers, increases electrical conductivity. Okra plant fibers are better candidates as lightweight conductive materials because of their lower densities and the moisture content in fibers increases conductivity [128,129, 4]. The results observed in this study give new feasibility for use of natural fiber and polymer composites as conductive filler in polymer. The results obtained help in the development of conducting polymer composites for medical applications such as electrodes will be suitable candidates for flexible, environmentally safe, lightweight and all-organic of energy storage devices [130,131].

Figure (4.13B,DandE) is evident that there exist a transition between two linear regions of the curve at (4.7)V. Figure (4.13B) reveals that, the I-V characteristics between (0.7-4.7)V, figure (4.13D) between (1.2-4.7)V and Figure (4.13E) between (1.4-4.2) V has been linear. Thus PANI and (PANI/ okra plant waste/0.5 and1% Ag) nanocomposites behaves as ohmic material within the region of 0.7 to 4.7 V. The second region of I-V characteristics lowest 0.7, 1.2 and 1.4 V thus, PANI and (PANI/okra plant waste/ 0.5 and 1% Ag) nanocomposites respectively has non-ohmic in nature. This property is greatly useful in the fabrication of rectifiers and photodiodes [132].

The current–voltage (I-V) in figure (4.13F) exhibits non-ohmic nature nature for (PANI/okra plants waste/1.5% Ag) nanocomposites. In PANI, polarans and bipolarans are produced due to doping of silver nanoparticals. The

non- ohmic behavior reveals that the contribution of charge transport is due to polarans and bipolarans in addition to free charge carriers (electrons and holes) [132].

Finger (4.13G) showed the I–V characteristic curve of (PANI/okra plant waste/2%Ag) nanocomposites thin film exhibit rectifying behavior (diode like). The I-V traces move from ohmic to semiconductor and perhaps the I-V curves (PANI/okra plants waste/2% Ag) nanocomposites display exponential increases [133, 134].
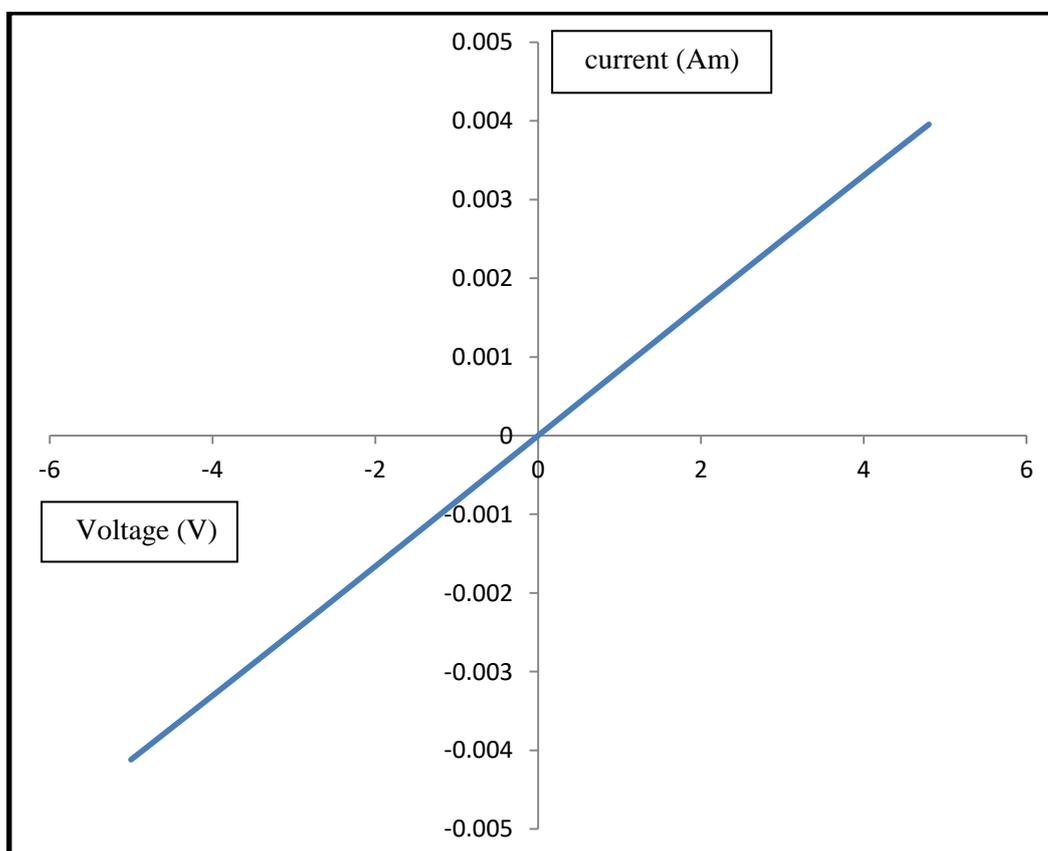
Figure (4.13A): Current-Voltage of okra plant.
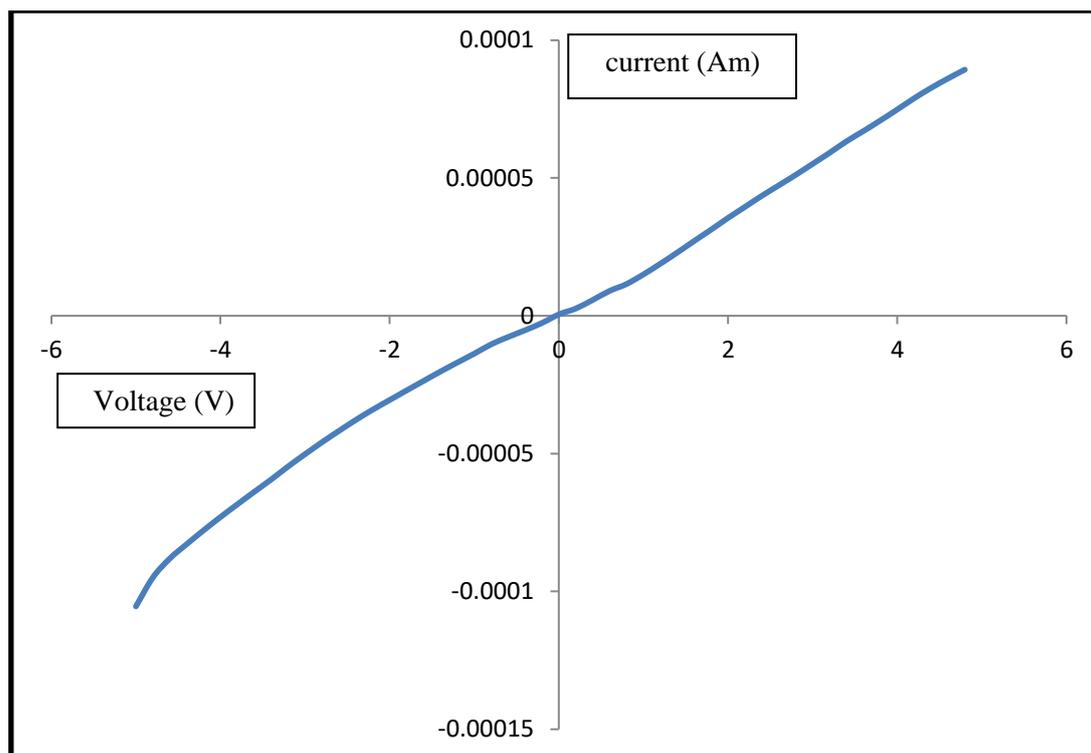
Figure (4.13B): Current-Voltage of PANI.
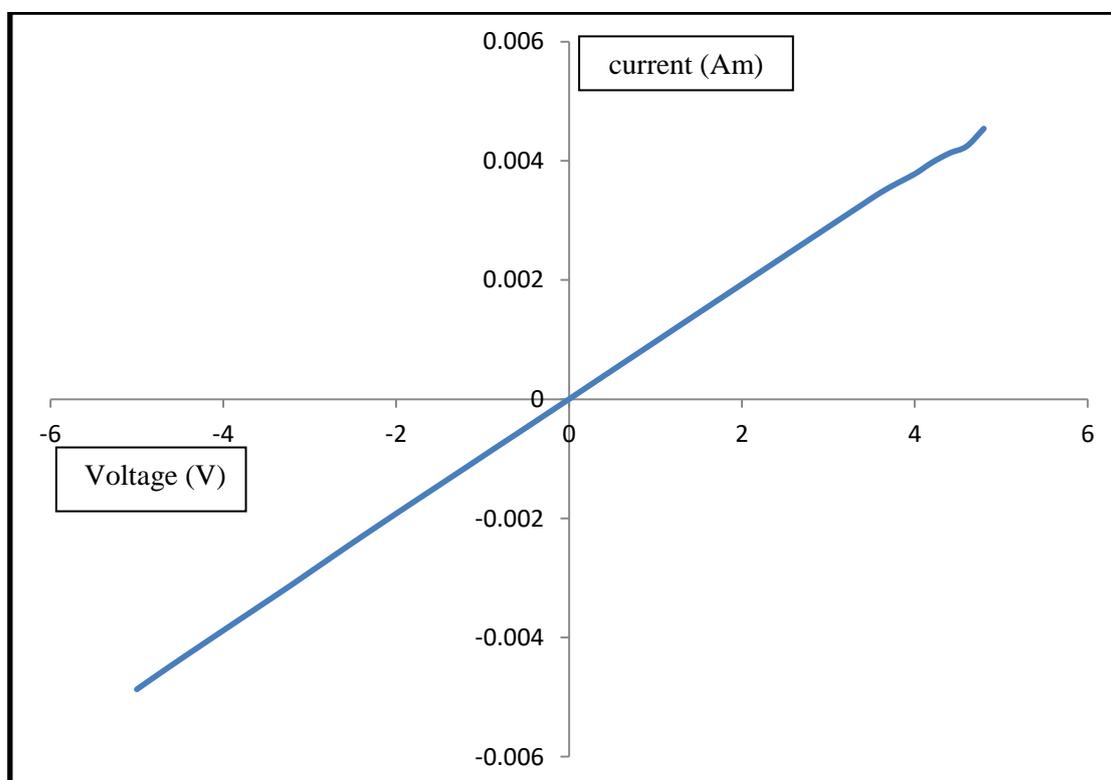
Figure (4.13C): Current-Voltage of (PANI/ okra plant waste) blend.
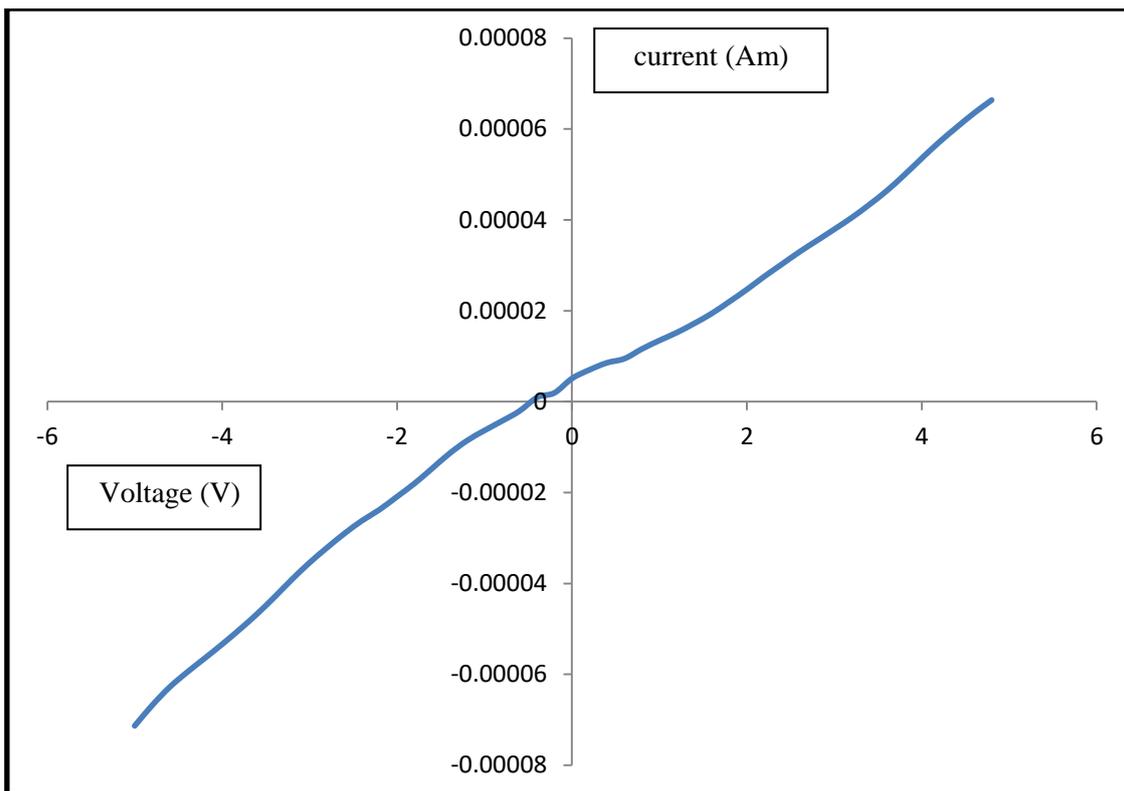
Figure (4.13D):Current-Voltage of (PANI/ okra plants waste/0.5% Ag) nanocomposites.
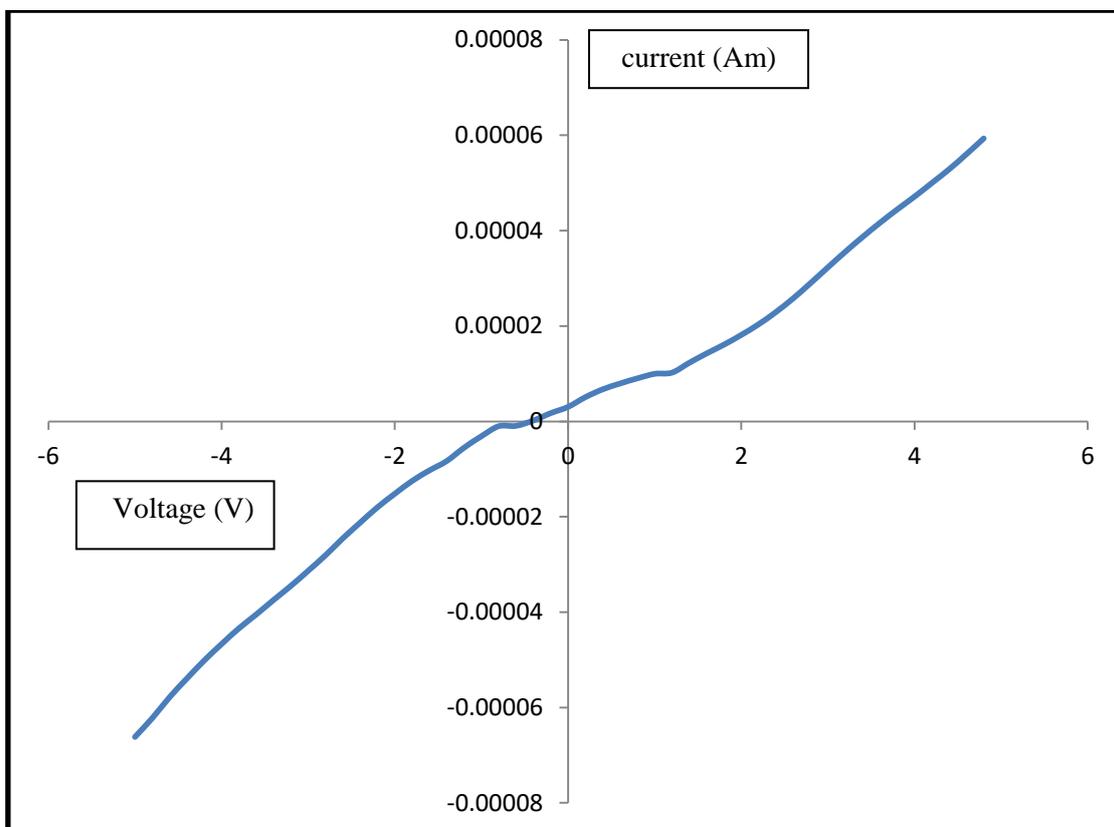
Figure (4.13E): Current-Voltage of (PANI/okra plants waste / 1% Ag) nanocomposite.
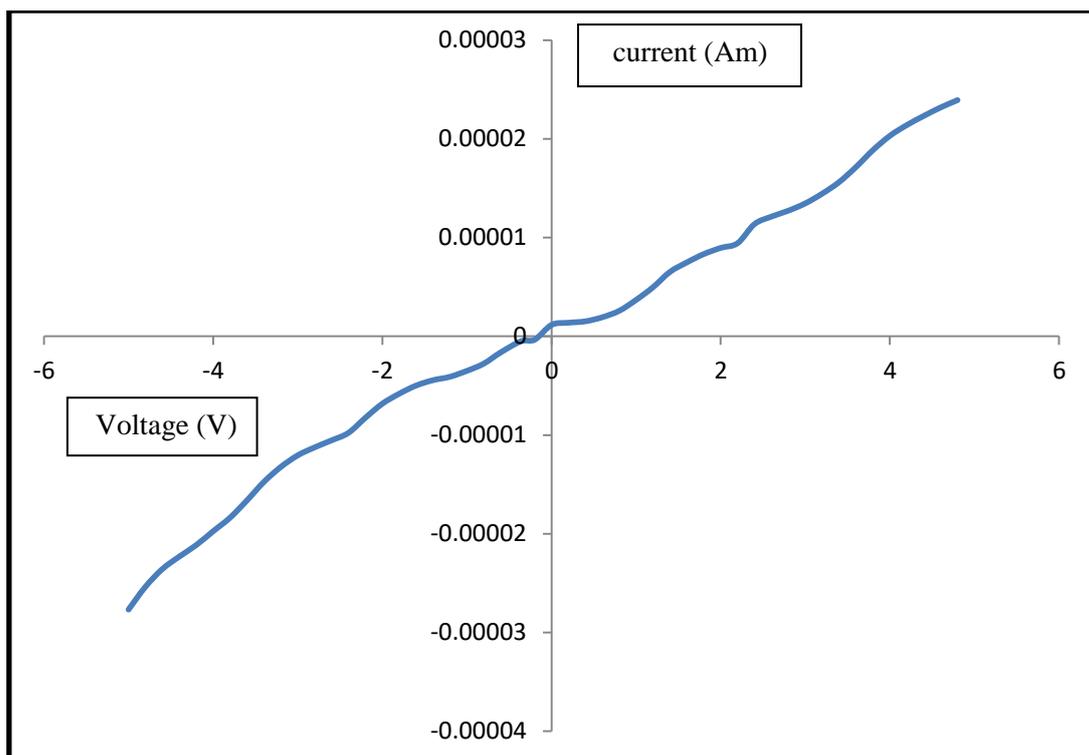
Figure (4.13F): Current-Voltage of (PANI/okra plants waste/ 1.5% Ag) nanocomposite.
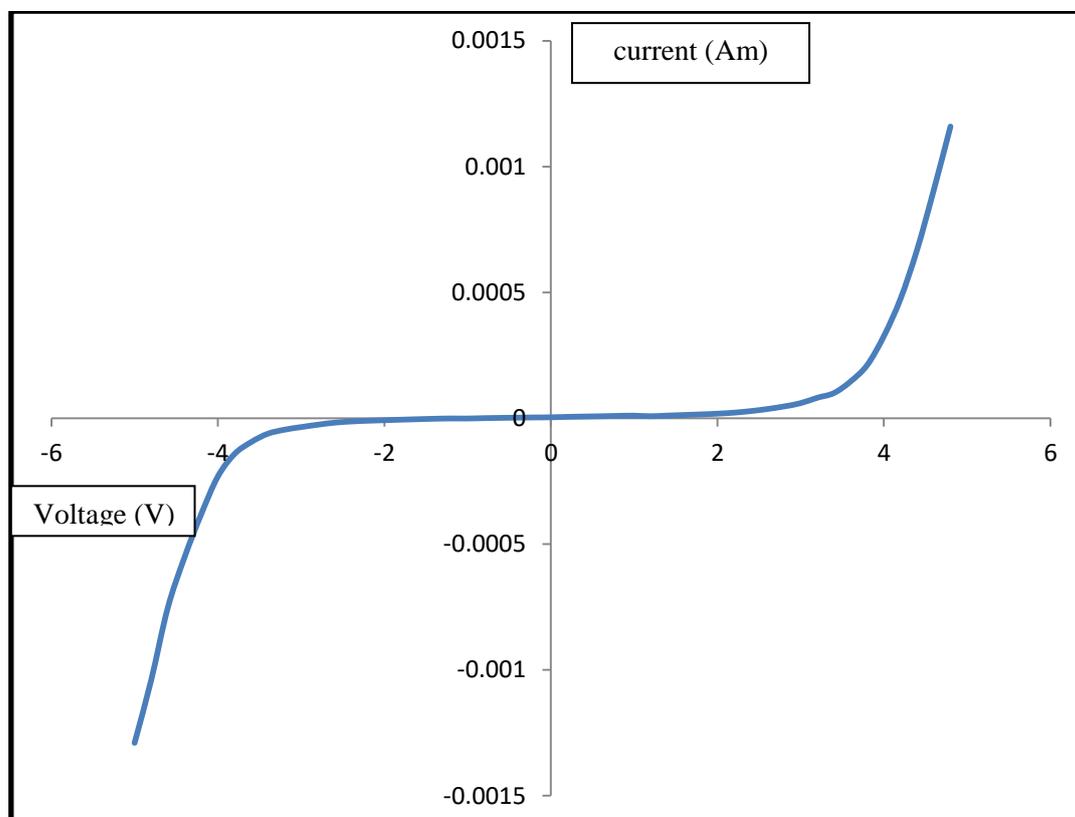
Figure (4.13G): Current-Voltage of (PANI/okra plants waste / 2% Ag) nanocomposite.

4.5 DC-Electrical Conductivity

Figure (4.14) show the DC electrical conductivity for each of (PANI/okra plant waste) blend, okra plants waste, PANI, and (PANI/okra plant waste/Ag) nanocomposites measured by Keithley electrometer type 2400. Where this Figure shows the effect of the okra plant on the electrical conductivity values in the temperature range (239-383)K for a of samples prepared. The results indicate that the electrical conductivity of the (PANI/okra plant waste) blend is higher than that of the (PANI/okra plant waste/Ag) nanocomposite. The electrical conductivity depends mainly on the temperature, which increases with increasing temperature to increase the number of charge carriers, from the above results we can conclude that the mobility mechanism is responsible for increasing the electrical conductivity [62].

From the figure (4.15) it is clear the relation between $\ln\sigma$ and the vs 1000/T of temperature for samples prepared. The values of activation energy calculated from figure (4.15) and are given in table (4.3). It was found that the activation energy for PANI and (PANI/okra plant waste/Ag) nanocomposites are higher than (PANI/okra plant waste) blend and okra plant waste. An increase in DC-conductivity with corresponding decrease in activation energy is found to be associated with a shift of Fermi level in the samples [62].

From a value of activation energy it is clear that the conduction is through the carrier concentration at the Fermi level [62]. Thus, adding the okra plant will host new energy levels within the band gap of the (PANI/okra plant waste) blend, reducing the energy gap and increasing the conductivity [135,120]. These results are disagreement with [62,76].
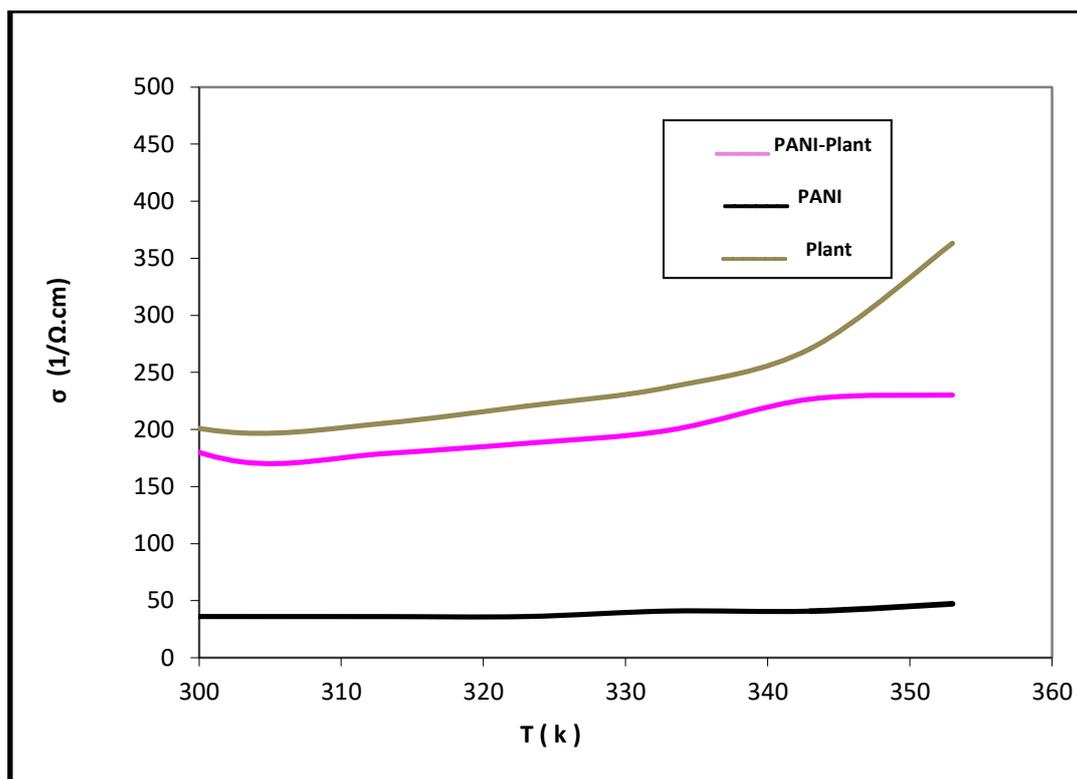
Figure (4.14A): Variation of (DC) conductivity with temperature of (PANI/okra plant waste) blend.
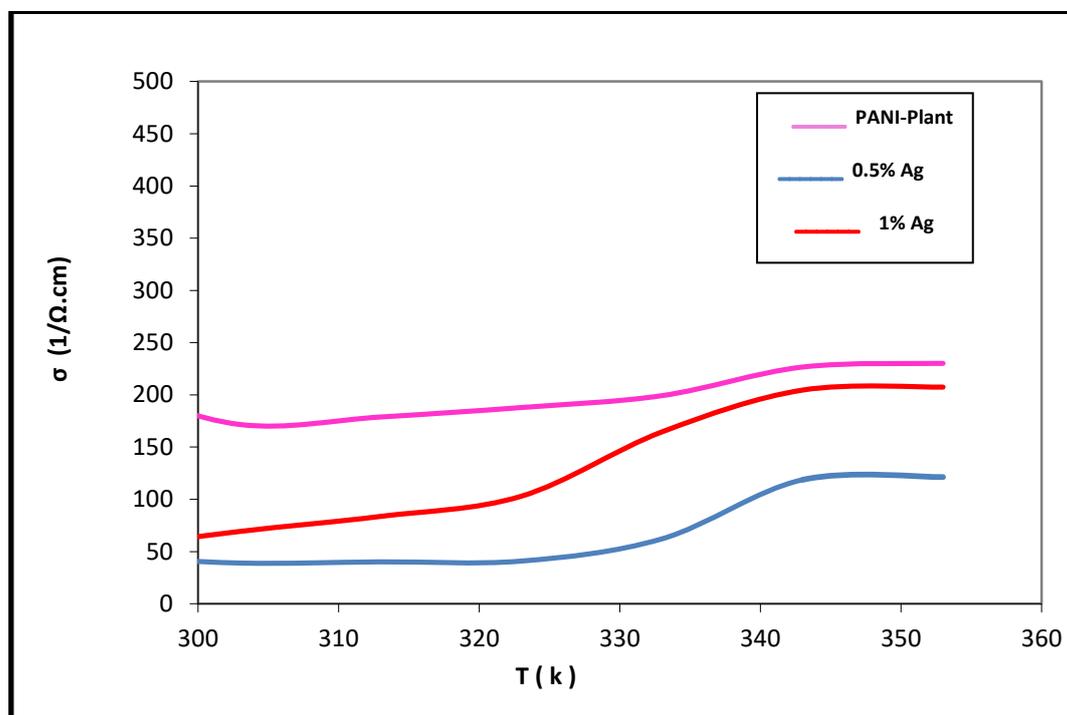
Figure (4.14B): Variation of (DC) conductivity with temperature of (PANI/ okra plant waste/ Ag) nanocomposites.
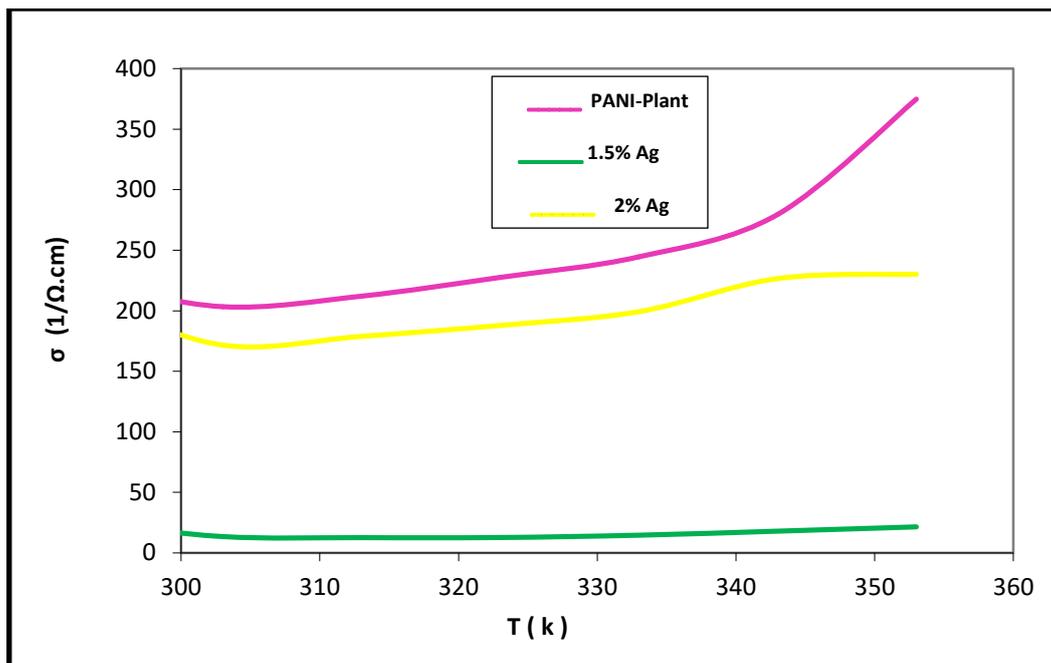
(C)

Figure (4.14C): Variation of (DC) conductivity with temperature of (PANI/ plants waste/ Ag) nanocomposites.
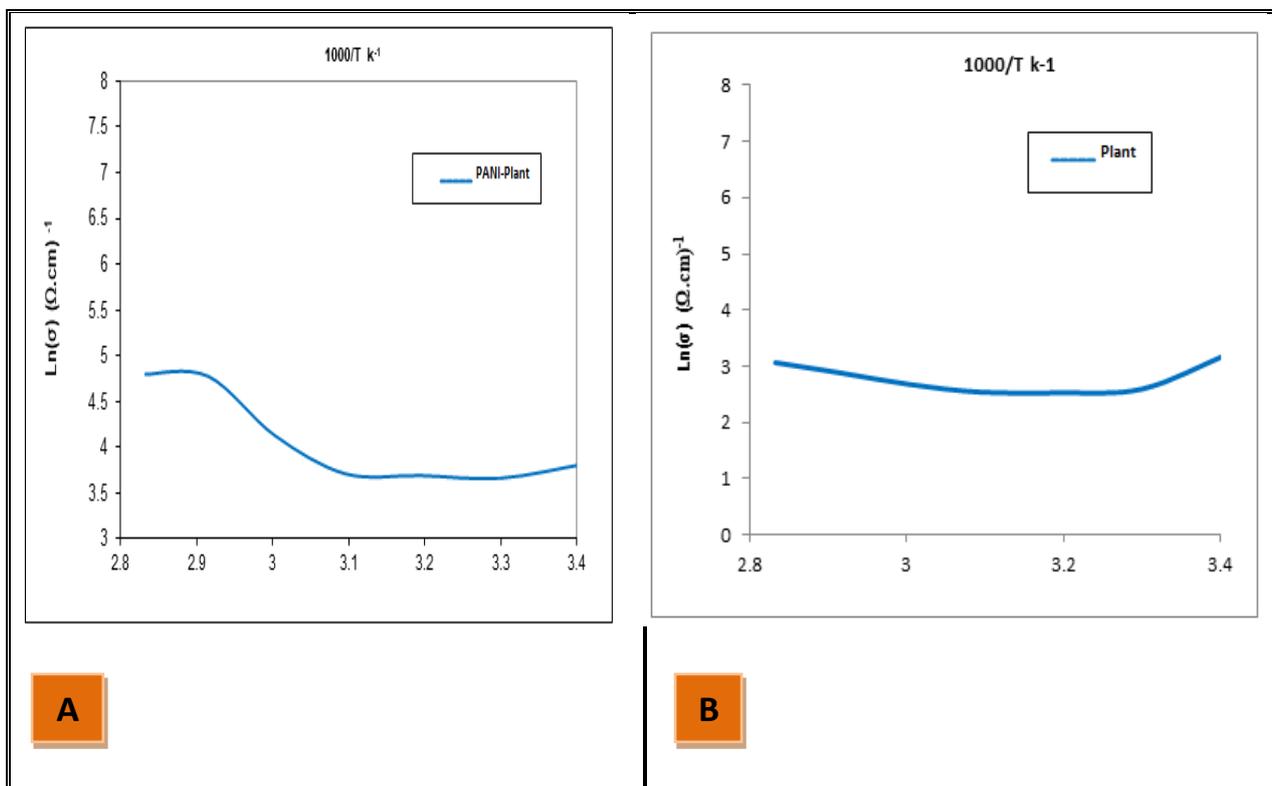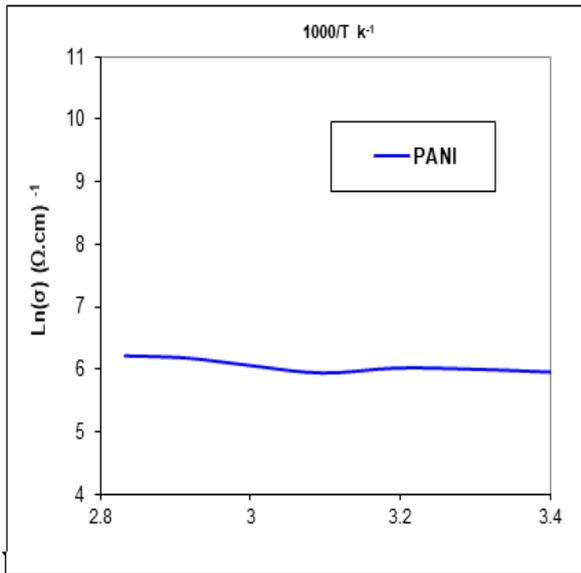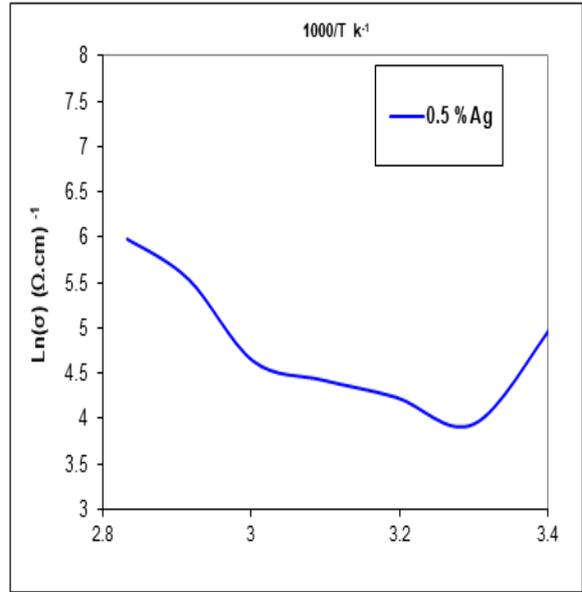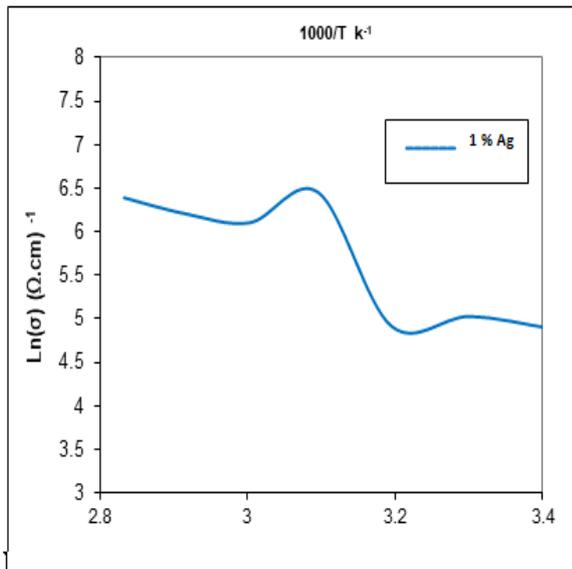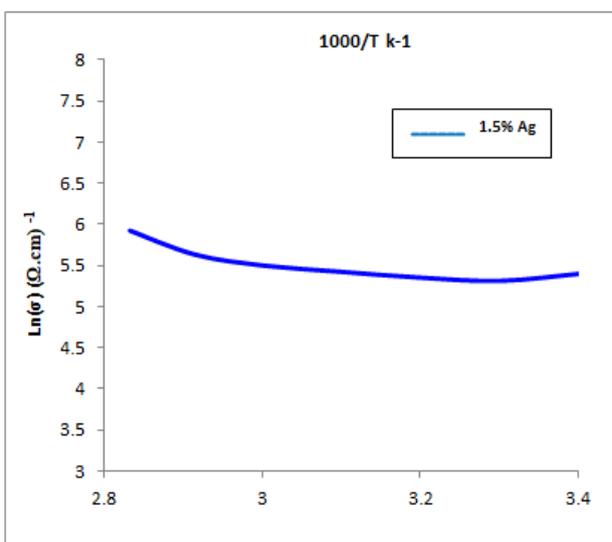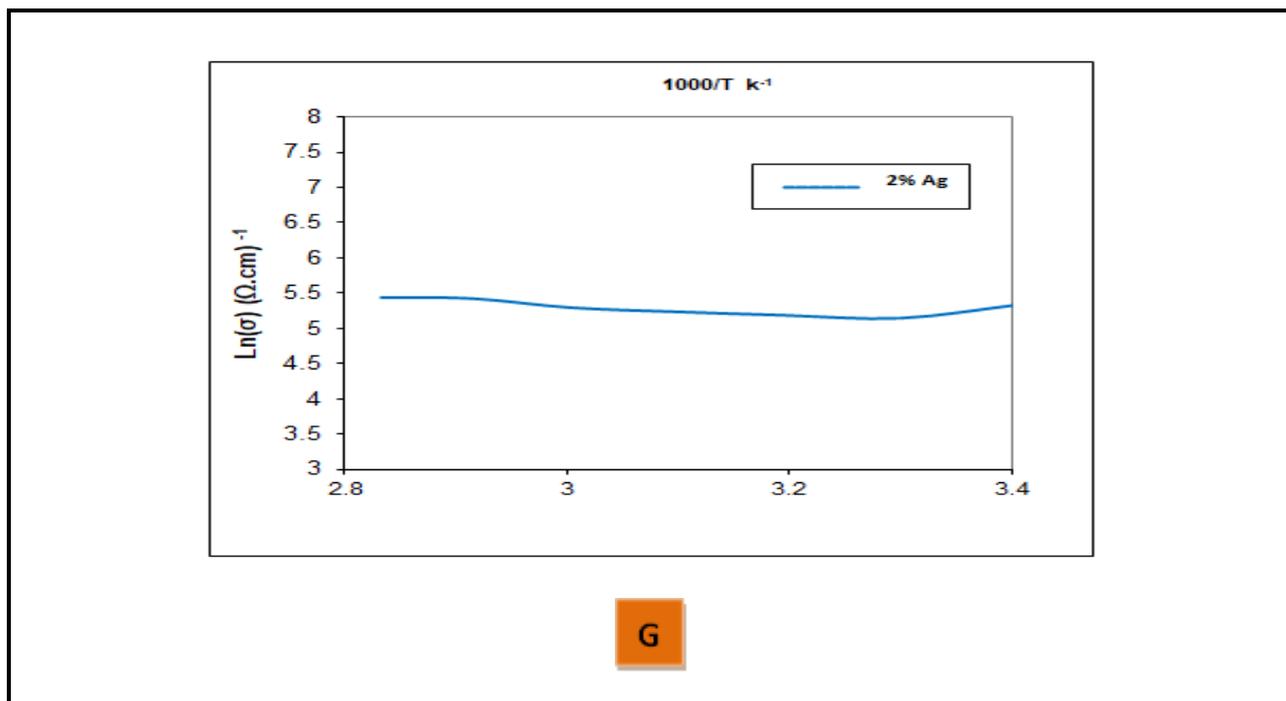
Figure (4.15): Ln(σ)versus 1000/T for (A) (PANI/okra plant waste) blend (B) okra plant (C) PANI and (PANI/ okra plant waste/ Ag) nanocomposites (D, E, F and G).

Table (4.3): Values of activation energy of (PANI/okra plant waste) blend and (PANI/okra plant waste/ Ag) nanocomposites.

Sample	Activation energy (Ea) eV
(PANI/okra plant waste) blend	0.0764
Okra plant	0.0738
PANI	0. 1432
(PANI/okra plant waste/ 0.5%Ag) nanocomposites	0.3024
(PANI/okra plant waste/1%Ag) nanocomposites	0.2709
(PANI/okra plant waste /1.5% Ag) nanocomposites	0.2181
(PANI/okra plant waste /2% Ag) nanocomposites	0.1555

4.6 Medical Applications

4.6.1 Antibacterial Activity

The antibacterial activity of (PANI/okra plant waste) blend   and (PANI/okra plant waste/Ag) nanocomposites was tested against two model pathogens Staphylococcus epidermidis as gram positive bacteria and Klebsiella pneumoniae as gram negative bacteria. The inhibitory activity was measured based on the diameter of the clear inhibition zone. If there was no surrounding clear zone, it was assumed that there was no inhibitory zone. The zones of inhibition around pieces of (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites for bacterial are shown in figure (4.16) while numerical values of inhibition zone diameter were compiled in table (4.4).

The results exhibited very high toxicity against gram negative bacteria, lower toxicity against gram positive bacteria. As shown in table (4.4) the films with okra plants presented reduction in cell viability of both Staphylococcus epidermidis and Klebsiella pneumoniae. These results indicated that (PANI/plants waste) blend were more effective against gram negative than the gram positive bacterium and found that films with Ag showed stronger antimicrobial activity against gram positive bacteria. The difference in the vulnerability of the bacteria could be related to the structure, and composition of the membrane cell wall between Staphylococcus epidermidis and Klebsiella pneumoniae Ag nanoparticales may directly bind with outer cell wall of gram positive bacteria which contains plenty of pores to make easy penetration of nanoparticles into the cells and thus causing leakage of intracellular contents and leads to cell death, but regarding gram negative bacteria, Ag nanoparticles bind primarily with outer cell membrane which comprises lipoprotein, lipopolysaccharide and phospholipids that may decrease the attachment of nanoparticles [136, 137].

The antibacterial activity of (PANI/okra plants waste) blend showed higher activity against the gram negative bacteria. okra plant amounts of vital nutrients like protein, calcium, iron and zinc and contain amount of antinutrients with high mineral bioavailability that components an important source for antibacterial activity affect bacterial growth of films containing okra mucilage. Okra mucilage is of polysaccharides correlate with antinutrient contents of the films it showed activity against Klebsiella pneumoniae, also it showed activity against Staphylococcus epidermidis. The presence of okra plants along with conductive polymer film and nanocomposites possessed inhibitory effect which inhibited growth of bacteria as foodborne pathogens. Based on our results, the fabricated films potential to be used as environment friendly antimicrobial packaging films [138]. These results are agreement with [70].
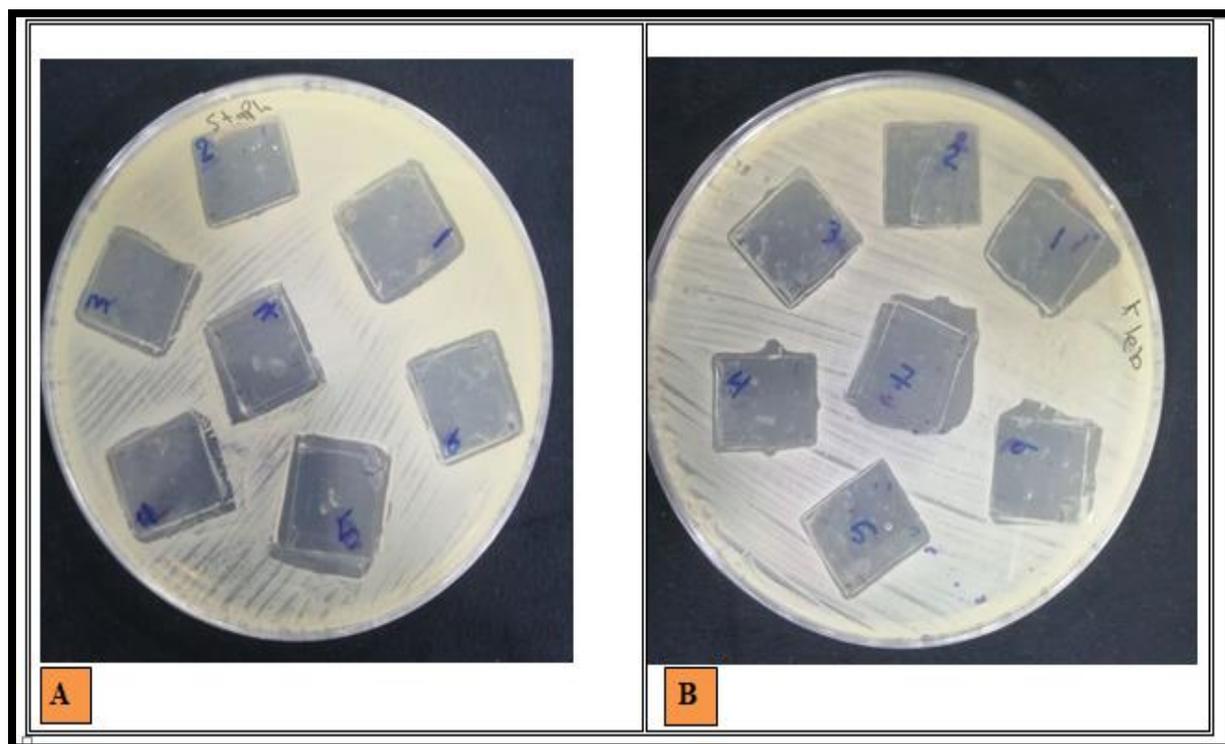
Figure (4.16): Antibacterial activity of (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites against (A) Staphylococcus epidermidis (B) Klebsiella pneumoniae.

Table (4.4): Antibacterial activity of (PANI/okra plant waste) blend and (PANI/okra plant waste/ Ag) nanocomposites against pathogen.

No.	samples	Inhibition Zone Diameter (mm)	
		Staphylococcus epidermidis	klebsiella pneumoniae
1	(PANI/ okra plant waste) blend	33	36
2	(PANI/ okra plant waste / 0.5%Ag) nanocomposites	31	32
3	(PANI/ okra plant waste / 1%Ag) nanocomposites	32	33
4	(PANI/ okra plant waste / 1.5%Ag) nanocomposites	33	34
5	(PANI/ okra plant waste / 2%Ag) nanocomposites	34	35
6	Okra plant	32	34
7	PANI	35	36

4.6.2 Electrodes

Electrodes was studied resistance polymer-based of the prepared samples for (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites by Keithley electrometer type 2400. Figure (3.17) display the behavior of resistance of the prepared samples for (PANI/ okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites at the temperature range (239-383)K. The figure showed that the resistance of (PANI/ okra plant waste) blend, okra plants waste decreases with increasing the temperature. The electrical resistance of (PANI/ okra plant waste) blend and okra plants waste are (3.75-1.13) KΩ   and (1.30-1.19) KΩ respectively it is less than of the

stratum corneum (SC). The SC is the upper skin layer (10–15μm in height) consisting of a stratified stratum, made of high-densely packaged cornified cells, called corneocites, which provide a very efficient electrical and chemical barrier electrical resistance of about 10^6 Ω, which characterizes human skin [139]. The SC is the mayor contributor to the skin resistance, with a resistance that ranges between (200) KΩ and (200) Ω for a square centimeter, although some authors state it to be up to 1 MΩ. Typical values of the electrode-skin resistance range between 150 to 200 KΩ and 5 to 10 KΩ before and after gel applications, respectively [140]. The result indicate that the sample of (PANI/plants waste) blend and okra plants waste are considered as the optimized sample show less resistance from PANI, and (PANI/okra plant waste/Ag) nanocomposites are shown in table (4.5). This result indicates that the presence of the okra plant with (PANI/okra plant waste) blend increased the polymer's conductivity, where is the okra plant as a filler for polymer matrices and contains a variety of organic reducing agents in various combinations and concentrations [120,141]. As shown in the tables (4.6), (4.7) and (4.8), the resistance decreases with the increase of the temperature.

The measurements of the resistance of (PANI/okra plant waste) blend (3.7-1.1) kΩ showed less than the resistance of the skin (SC) and the electrode-skin resistance of three types of bioelectrodes (wet, semi-dry, and dry)The electrode-skin resistance always decreases at various skin locations all electrodes. The electrode resistance of the dry electrode, semi-dry electrodes, and wet electrode are (57.5–540.0) kΩ, (10.3–38.4) kΩ and (1.4–2.8) kΩ respectively [140], thus (PANI/okra plant waste) blend prepared by thin films method can be used to manufactured electrode a low-cost, appearing easy to use and to apply by (PANI/okra plant waste) blend to recording ECG, EMG and EEG signals. Where are monitoring biosignals

ECG, EMG and EEG is important for a better understanding of the pathological and physiological conditions of human subjects.
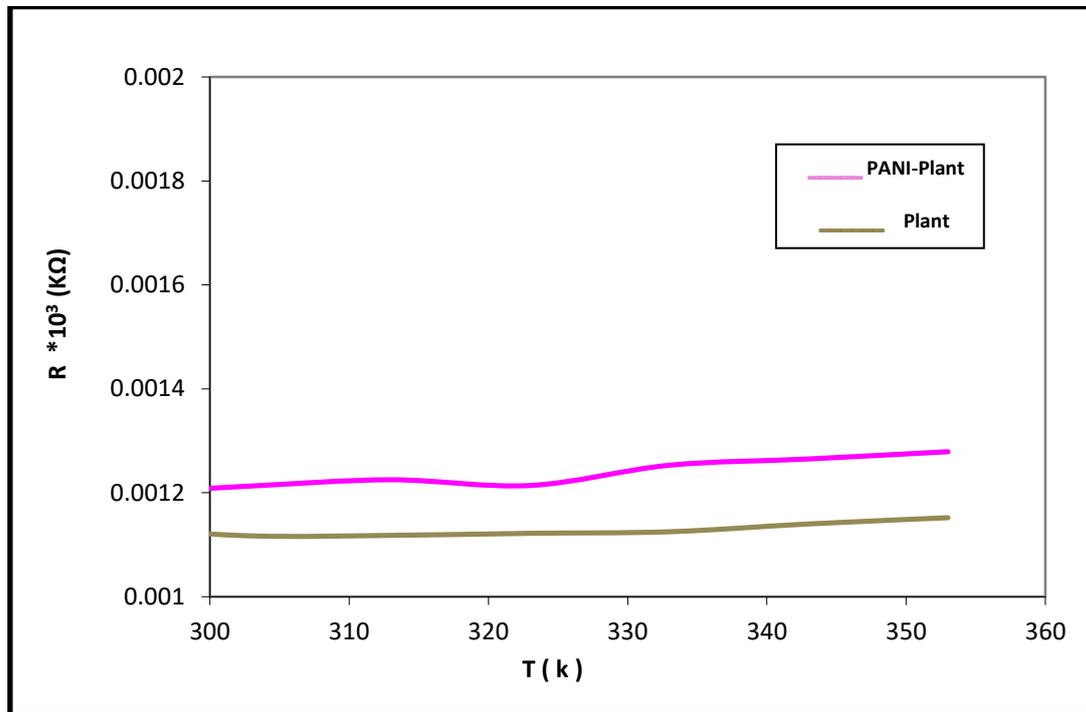
(A)
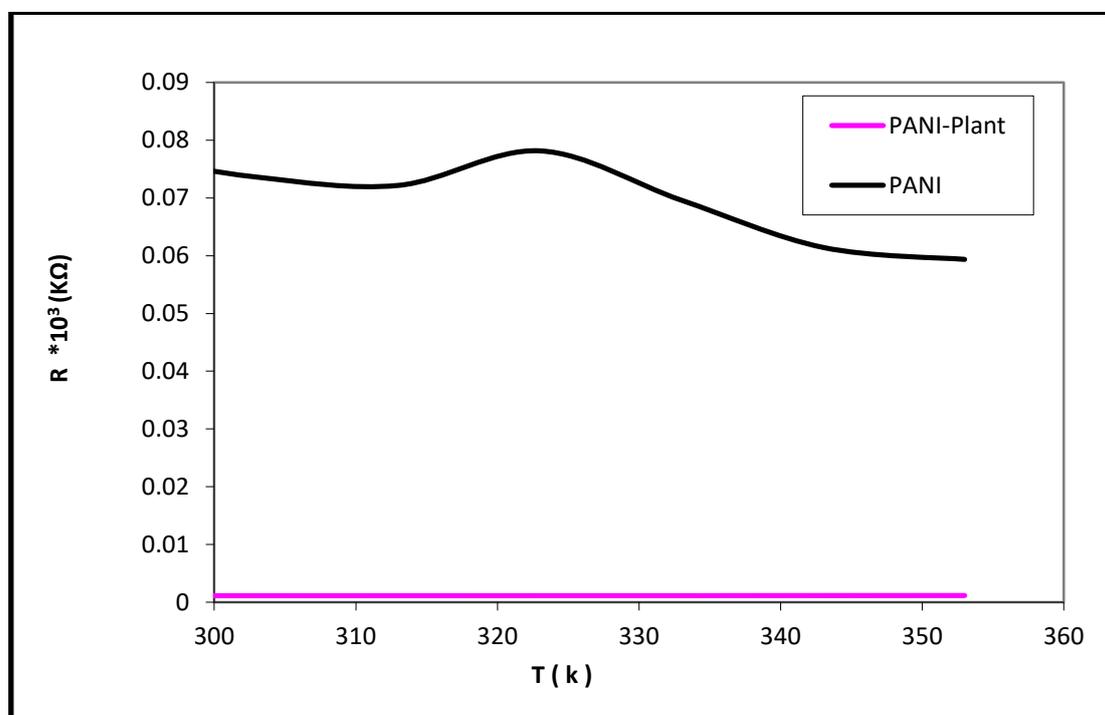
(B)

Figure (4.17): Resistance of (A and B) (PANI/okra plant waste) blend
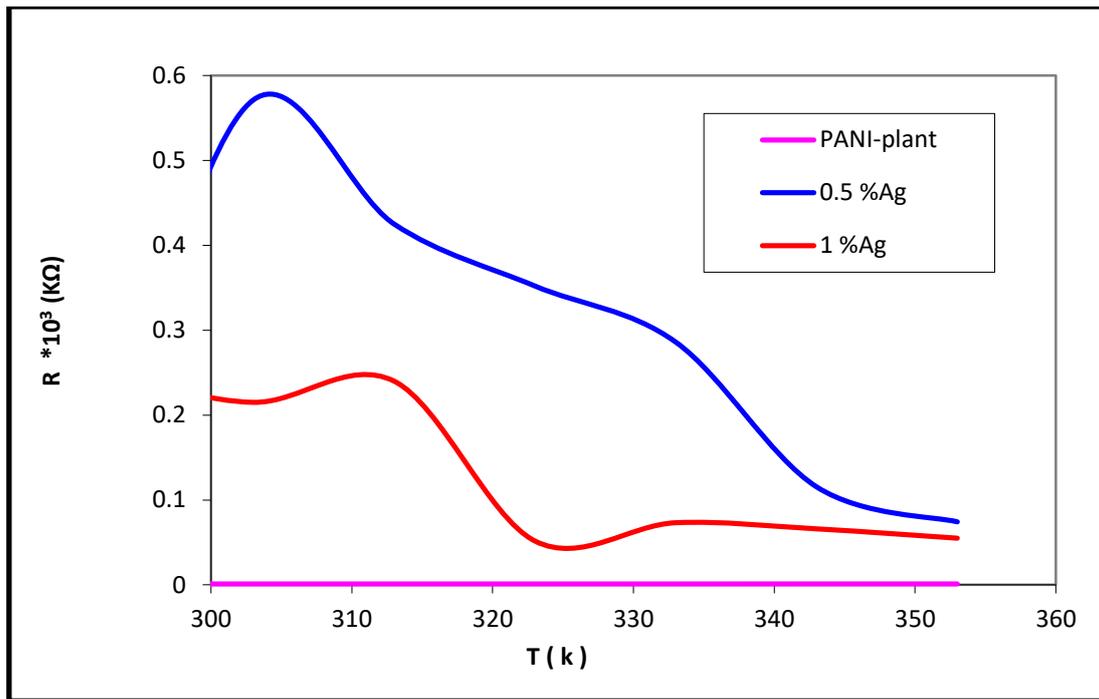
(C)
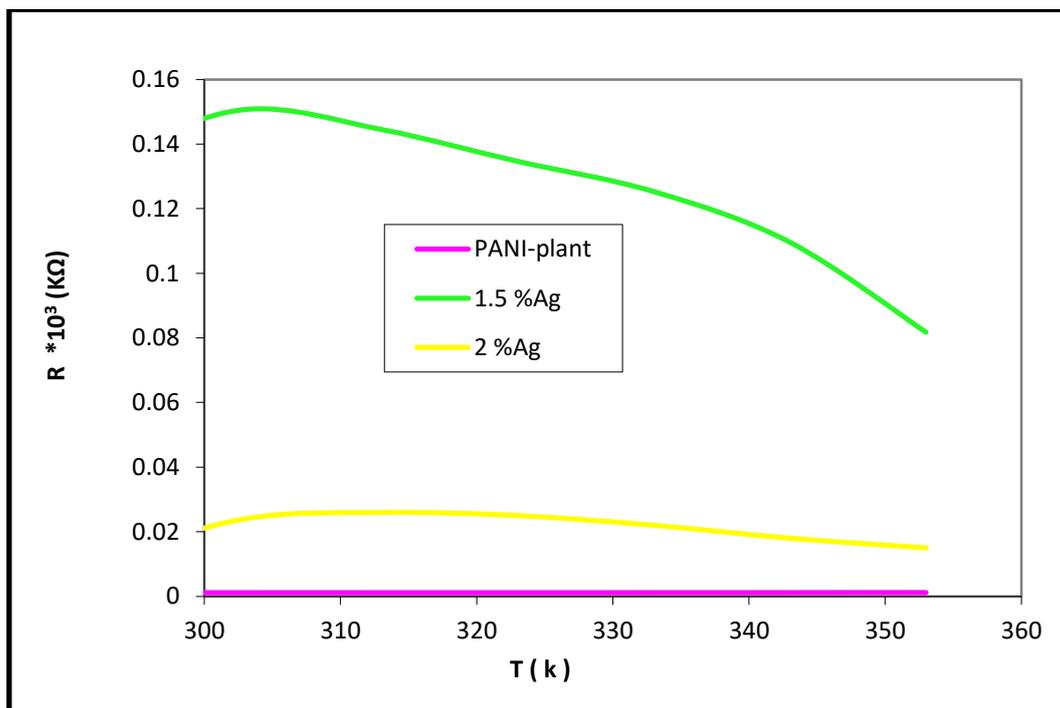
(D)

Figure (4.17): Resistance of (C and D) (PANI/okra plant waste/ Ag) nanocomposites.

Table (4.5): Resistance of (PANI/ okra plant waste) blend and (PANI/ plants waste/ Ag) nanocomposites.

Sample	Resistance kΩ
(PANI/okra plant waste) blend	(3.75-1.13)
Okra	(1.30-1.19)
PANI	(77.52-25.54)
(PANI/okra plant waste/0.5% Ag) nanocomposites	(179.2-31.14)
(PANI/okra plant waste/1% Ag) nanocomposites	(247-38.55)
(PANI/okra plant waste/ 1.5%Ag) nanocomposites	(136.71-39.22)
(PANI/okra plant waste/2% Ag) nanocomposites	(24-12)

Table (4.6):Values of resistance of (PANI/ okra plant waste) blend and (PANI/ plants waste/ Ag) nanocomposites at T = 293 K.

Sample	Resistance kΩ
(PANI/ okra plant waste) blend	3.75
Okra	1.198
PANI	77.52
(PANI/okra plant wast /0.5% Ag) nanocomposites	179.2
(PANI/okra plant waste/1% Ag) nanocomposites	247
(PANI/okra plant waste /1.5%Ag) nanocomposites	144.52
(PANI/ plants waste/2% Ag) nanocomposites	26

Table (4.7):Values of resistance of (PANI/ okra plant waste) blend and (PANI/ plants waste/ Ag) nanocomposites at T = 313 K.

Sample	Resistance kΩ
(PANI/ okra plant waste) blend	3.66
Okra	1.25
PANI	72.61
(PANI/okra plant waste/0.5% Ag) nanocomposites	425
(PANI/ okra plant waste/1%Ag) nanocomposites	240
(PANI/ okra plant waste/1.5%Ag) nanocomposites	136.71
(PANI/ okra plant waste/2% Ag) nanocomposites	24

Table (4.8):Values of resistance of (PANI/ okra plant waste) blend and (PANI/ plants waste/ Ag) nanocomposites at T = 343 K.

Sample	Resistance kΩ
(PANI/okra plant waste) blend	1.11
Okra	1.21
PANI	61.43
(PANI/okra plant waste /0.5% Ag) nanocomposites	115.54
(PANI/ okra plant waste / 1%Ag) nanocomposites	55
(PANI/ okra plant waste / 1.5%Ag) nanocomposites	109.63
(PANI/ okra plant waste /2% Ag) nanocomposites	18

4.6.3 Polymer Eyeglasses Lens

as in the Figure (4.6AandB) presents a graph of electromagnetic radiation transmittance as a function of the wavelength from (300-1100) nm .The average transmittance value between (400) nm and (800) nm for the (PANI/plants waste) blend, okra plants waste and PANI were (%0.961, 0.980 and 0.967) respectively and (0.985%, 0.980%, 0.977% and 0.987%) for the (PANI/okra plant waste/0.5%, 1%, 1.5% and 2% Ag) nanocomposites respectively. The determined values UVR region and visible region are shown in table (4.9). The most important criteria for the selection of optical polymer for polymer lenses applications eyeglasses lens is high transmittance (T) of incident light in the visible light range. Generally, all eye glasses should feature a high transmittance value between 400 nm and 800 nm within the total visible range, the transmittance value at wavelengths after (400) nm should be more than 85% on visible-light ranges [142].

In the wavelength range from about (320 – 400) nm, there is a very large increase in light transmission. The research concerns the transmission of electromagnetic waves with a wavelength corresponding to the visible part of the spectrum and part of the ultraviolet and infrared radiation range and the obtained results do confirm about the increase in light transmittance (PANI/okra plant waste) blend and nanocomposites in the range of wavelengths corresponding to the highest sensitivity of the human eye [142,143]. The use of plants waste (okra plant) generates effect in the optical properties of the allowing the increase of its transparency (PANI/okra plant waste) blend and nanocomposites and obtaining of systems with adequate dispersions and distributions of the plants waste in the PANI matrix. As a result the prepared polymer from plants waste will create a new territory in polymer eyeglasses lens industry. These results are in agreement with [142].

Table (4.9): Transmittance value of (PANI/ okra plant waste) blend and (PANI/ okra plant waste / Ag) nanocomposites.

Wave Length(nm)	Transmittance %						
	(PANI/okra plant waste) blend	Okra plant waste	PANI	(PANI/okra plant waste/Ag) nanocomposites			
				0.5% Ag	1% Ag	1.5% Ag	2% Ag
400-800	0.961	0.980	0.967	0.985	0.980	0.977	0.987
376	0.959	0.976	0.975	0.991	0.981	0.975	0.977
403	0.963	0.980	0.976	0.996	0.985	0.975	0.978
483	0.961	0.970	0.976	0.991	0.982	0.978	0.978
704	0.961	0.980	0.978	0.980	0.979	0.976	0.978
723	0.961	0.980	0.977	0978	0.978	0.977	0.978
756	0.959	0.980	0.977	0.978	0.977	0.977	0.978

4.6.4 Gradient-Refractive Index Lens (GRIN)

Refractive index of the prepared films for (PANI/plants waste) blend and (PANI/plants waste/silver) nanocomposites was studied. It should be noted that the refractive index in the resulting of the prepared samples are (1.131) in (PANI/plants waste) blend and (1.048, 1.065, 1.077 and 1.073) in (PANI/plants waste/0.5%, 1%, 1.5% and 2% / Ag) nanocomposites respectively, these results are plotted as a function of wavelength film in figure (4.8). The refractive index of the GRIN lens varies from (0.451) to (1.719) [144]. The results indicated that the refractive index of (PANI/okra plant waste) blend and nanocomposites was sufficient to demonstrate satisfactory optical performance in the GRIN lens. These results are in agreement with [144]. The determined average values

refractive index are shown in table (4.10). The values of the refractive index for the prepared (PANI/okra plant waste) blend (PANI/okra plant waste/Ag) and nanocomposites at $\lambda = $ (400, 600 and 700) nm are shown in tables (4.11), (4.12) and (4.13) respectively.

Table (4.10): The average values refractive index of (PANI/okra plant waste) blend and (PANI/okra plant waste/ Ag) nanocomposites.

Sample	Refractive Index
(PANI/ okra plant waste) blend	1.131
Okra plants	1.066
PANI	1.075
(PANI/okra plant waste / 0.5%Ag) nanocomposites	1.048
(PANI/okra plant waste/ 1%Ag) nanocomposites	1.065
(PANI/ okra plant waste /1.5% Ag) nanocomposites	1.077
(PANI/okra plant waste /2% Ag) nanocomposites	1.073

Table (4.11): Refractive index value of (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites at $\lambda = $ 400 nm.

Sample	Refractive Index
(PANI/okra plant waste) blend	1.125
Okra plant	1.066
PANI	1.079
(PANI/okra plant waste /0.5%Ag) nanocomposites	0.945
(PANI/okra plant waste /1%Ag) nanocomposites	1.048
(PANI/ okra plant waste /1.5% Ag) nanocomposites	1.079
(PANI/okra plant waste /2% Ag) nanocomposites	1.073

Table (4.12): Refractive index value of (PANI/okra plant waste) blend and (PANI/ okra plant waste / Ag) nanocomposites at λ = 600 nm.

Sample	Refractive Index
(PANI/okra plant waste) blend	1.127
Okra plant	1.066
PANI	1.074
(PANI/okra plant waste/ 0.5%Ag) nanocomposites	0.989
(PANI/okra plant waste/ 1%Ag) nanocomposites	1.064
(PANI/ okra plant waste /1.5% Ag) nanocomposites	1.074
(PANI/okra plant waste /2% Ag) nanocomposites	1.073

Table (4.13): Refractive index value of (PANI/okra plant waste) blend and (PANI/ okra plant waste/Ag) nanocomposites at λ = 700 nm.

Sample	Refractive Index
(PANI/okra plant waste) blend	1.130
Okra plant	1.066
PANI	1.074
(PANI/ okra plant waste/ 0.5%Ag) nanocomposites	1
(PANI/okra plant waste/ 1%Ag) nanocomposites	1.069
(PANI/okra plant waste/1.5% Ag) nanocomposites	1.074
(PANI/ okra plant waste /2% Ag) nanocomposites	1.073

4.7 Conclusions

In our study, the following results were obtained:

1- The absorbance, absorption coefficient, extinction coefficient, imaginary dielectric constants and optical conductivity increase while transmittance and energy gap decrease with additive okra plant to polymer for (PANI/okra plant waste) blend.

2- D.C electrical conductivities of (PANI/okra plant waste) blend and (PANI/okra plant waste/Ag) nanocomposites were studied as a function of okra plant content of films, and the conductivity increased with okra plant content, furthermore the okra plant was found to be a more effective than Ag nanoparticals and effective reducing agent .

3- The morphological measurements have shown that the (PANI/okra plant waste) blend has a fine and grainy structure fused together and (PANI/okra plant waste/Ag) nanocomposites exhibit varying microstructures. The results confirm that these okra plant fibres show potential as reinforcement in polymer matrix blend.

4- For medical application the polymer eyeglasses lens and (GRIN) lenses, the samples showed a high ultraviolet (UV) blocking ratio in (400 – 800) nm. The results showed that the use of these okra plant waste in polymeric material leads to obtaining materials with a better refractive and improve transparency. In this way, the materials developed using okra plant waste of fillers and present highly promising for the development of higher transparent lenses.

5- For electrode application, the electrical resistance decrease with (PANI/okra plant waste) blend more than nanocomposites therefore these findings provide insights for the development of new electrodes to recording electrocardiography (ECG),electromyography (EMG) and electroencephalography (EEG) signals, because of the availability of okra

plant extract, the method used in this synthesis can be adopted for large-scale synthesis. Finally, we concluded that okra plant waste has showed better structural and electrical properties than Ag nanoparticals.

6- (PANI/okra plant waste) blend exhibited higher inhibition zone diameter for gram negative bacteria and lower inhibition zone diameter for gram positive bacteria and found that films with Ag showed stronger antimicrobial activity against gram positive bacteria. Okra plants was good antimicrobial agent.

4.8 Suggestions for Future Work

1- Preparation of conductive polymer prepared by solution casting method.

2- Studying the influence of okra plant on the mechanical and thermal properties of (PANI/ plants waste) blend and (PANI/ plants waste /Ag) nanocomposites.

3- Using Eggplant (Solanum melongena L.) plant to prepared conductive polymer.
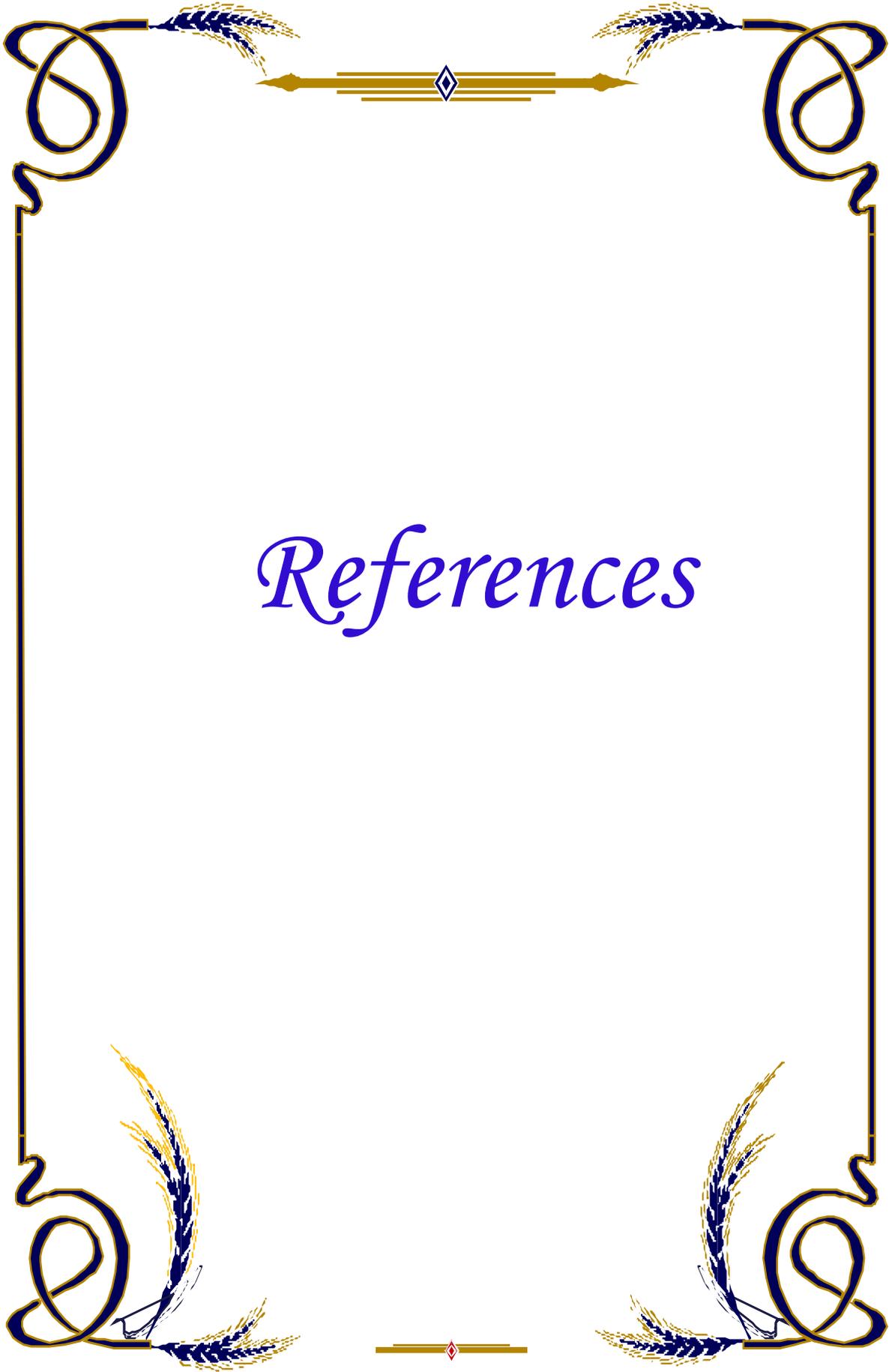
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الخلاصة

في هذا العمل تم تصنيع متراكبات صديقة للبيئة مكونة من البولي انيلين (PANI) ومستخلص نبات الباميا (okra plant waste) باستخدام طريقة الأغشية الرقيقة بواسطة Spin Coating. العينات المحضرة خليط (PANI/okra plant waste) و okra plant و PANI و المركبات النانوية (PANI/okra plant waste/Ag). تم اضافة جزيئات الفضة النانوية (Ag) إلى خليط (PANI/okra plant waste) بالنسب (0.5%, 1%, 1.5% and 2%). تمت دراسة الخواص التركيبية والكهربائية والبصرية للاغشية المحضرة وتطبيقاتها كأقطاب كهربائية و عدسات النظارات البوليمرية و عدسات الانكسار المتدرج (GRIN) والنشاط المضاد للبكتيريا. تمت دراسة الخصائص البصرية في مدى الطول الموجي nm (300-1100) و بينت النتائج أن الخواص البصرية لخليط (PANI/okra plant waste) تم تحسينها بإضافة نبات الباميا. بينت النتائج أن الامتصاصية تزداد في خليط (PANI/okra plant) كما تم حساب معامل الامتصاص وفجوة الطاقة الضوئية ، وأظهرت النتائج أن البوليمر يمتلك فجوة طاقة غير مباشرة مسموح بها وأنه يتناقص بإضافة نبات الباميا لتكوين المستويات الموضعية في فجوة الطاقة. تم قياس الخواص الكهربائية لخليط (PANI/okra plant waste) والمركبات النانوية و بينت النتائج أن الموصلية الكهربائية تزداد مع زيادة درجة الحرارة وأيضاً بإضافة نبات الباميا.

أظهرت نتائج تطبيق خليط (PANI/okra plant waste) كأقطاب كهربائية إلى انخفاض المقاومة الكهربائية وزيادة الموصلية مع plants waste المضافة إلى البوليمر. توفر هذه النتائج رؤى لتطوير أقطاب كهربائية جديدة لتسجيل تخطيط كهربائية القلب (ECG) ، تخطيط كهربائية العضلات (EMG) وتخطيط كهربائية الدماغ (EEG). تُظهر نتائج تطبيقات عدسات النظارات البوليمرية وعدسات معامل الانكسار المتدرج (GRIN) لخليط (PANI/okra plant waste) والمركبات النانوية أنه في سلوكهم البصري لديهم انكسار أفضل وتحسين في الشفافية. وبالتالي، فإن استخدام نفايات النبات على أساس المواد البوليمرية ينتج عنه خصائص مختلفة في المواد المحضرة من نفايات النباتات ، عند استخدامها في تطبيقات عدسات البوليمر.

تم اختبار الخصائص المضادة للبكتيريا للعينات المحضرة ضد مسببات الأمراض Staphylococcus epidermidis كبكتيريا موجبة الجرام و klebsiella pneumoniae كبكتيريا سالبة الجرام. قدم خليط (PANI/okra plant waste) و المركبات النانوية نشاطًا مضادًا للبكتيريا ضد البكتيريا المختبرة. أظهرت الأغشية المحتوية على نبات الباميا نشاطًا أكثر كمضاد للبكتيريا، كانت الأنشطة المثبطة للأغشية الناتجة أقوى ضد klebsiella pneumoniae.

جمهورية العراق

وزارة التعليم العالي والبحث العلمي

جامعة بابل/ كلية العلوم

قسم الفيزياء

تصنيع متراكبات صديقة للبيئة مكونة من البولي انيلين ومستخلص نبات الباميا لتطبيقات الطب الحياتي

اطروحة

مقدمة إلى مجلس كلية العلوم- جامعة بابل

وهي جزء من متطلبات نيل درجة الدكتوراه فلسفة في العلوم /الفيزياء

من قبل

نورس حفظي شليوح شاكر

بكالوريوس علوم فيزياء/ ٢٠٠٦

ماجستير علوم فيزياء/ ٢٠١٩

بإشراف

أ.د. نهاد عبد الامير صالح خضير

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