UV-VIS STUDIES ON REDUCED GRAPHENE OXIDE SILVER NANOCOMPOSITE

KESHVINI A/P NARENDRAN
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DECLARATION

I declare that this thesis entitled Uv-Vis studies on reduced graphene oxide silver nanocomposite is the results of my own research except as citedin the references.

Signature :________keshvini_____________________

Student's Name :_____KESHVINI A/P NARENDRAN________________________

Date :__________18/1/2024__________________

Verified by:

Signature : ______________________________

Supervisor's Name :___Prof Madya DR Anamt Mohamed Noor____________________________

Stamp : ______________________________

Date : ______________________________

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ABSTRAK

Pengintegrasian Grafin Oksida yang direduksi (rGO) dengan nanopartikel silver dalam struktur nanokomposit merupakan suatu pendekatan yang menarik untuk memajukan bahan dengan sifat optik yang unik. Tujuan dari usaha penelitian ini adalah untuk mengembangkan cara sintesis komprehensif bagi nanokomposit GO-Ag, menganalisis perilaku optiknya, dan mencirikan atribut struktural dan morfologisnya. Dalam kaedah ini, nanokomposit GO-Ag disintesis melalui sintesis mikrogelombang. Kaedah analitis seperti spektroskopi UV-VIS, difraksi sinar-X (XRD), dan spektroskopi inframerah transformasi Fourier (FTIR) digunakan selama pencirian fasa untuk memastikan pencirian kimia dan struktural nanokomposit GO-Ag yang disintesis. Dengan menggunakan spektroskopi UV-VIS, sifat optik nanokomposit GO-Ag juga diselidiki secara mendalam. Pemeriksaan difokuskan pada menjelaskan bagaimana nanokomposit menyerap dan mentransmisikan cahaya di wilayah ultraviolet dan terlihat, sehingga memberikan informasi berharga mengenai sifat optiknya. Sintesis nanokomposit GO-Ag berhasil dilakukan, sebagaimana ditunjukkan oleh analisis morfologis dan struktural, yang mengungkapkan deposisi nanopartikel perak dalam tersebar yang seragam pada grafin oksida yang direduksi. Sifat optik nanokomposit diungkapkan melalui penyelidikan spektroskopi UV-VIS, yang menunjukkan pencirian penyerapan. Pemeriksaan menyeluruh yang dilakukan dalam penelitian ini meningkatkan pengetahuan mengenai karakteristik optik nanokomposit grafin oksida-perak.

Kata kunci: Spektroskopi UV-VIS, Difraksi Sinar-X (XRD), Spektroskopi Inframerah Transformasi Fourier (FTIR), nanokomposit grafin oksida-argentum, sintesis gelombang mikro.

ABSTRACT

The integration of ultraviolet-visible (UV-vis) reduced graphene oxide (rGO) with silver nanoparticles in a nanocomposite structure represents a compelling avenue for advancing materials with unique optical properties.The objective of this research endeavour was to develop a comprehensive synthesis method for the GO-Ag nanocomposite, analyse its optical behaviour, and characterise its structural and morphological attributes.For method, The GO-Ag nanocomposite was synthesised via microwave synthesis. Analytical methods such as UV-VIS spectroscopy , X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR) were employed during the characterization phase to ascertain the chemical and structural characteristics of the GO-Ag nanocomposite that was synthesised.Using UV-VIS spectroscopy, the optical properties of the GO-Ag nanocomposite were also investigated in depth. The examination was centred on clarifying the manner in which the nanocomposite absorbed and transmitted light in the ultraviolet and visible regions, thereby offering valuable information regarding its optical properties.The synthesis of the GO-Ag nanocomposite was accomplished successfully, as indicated by the morphological and structural analyses, which disclosed the deposition of silver nanoparticles in a uniform dispersion on reduced graphene oxide. The optical properties of the nanocomposite were revealed through UV-VIS spectroscopic investigations, which demonstrated its absorption characteristics.The through examination conducted in this study enhances knowledge regarding the optical characteristics of graphene oxide-silver nanocomposites.

Keywords:UV-VIS Spectroscopy, X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), graphene oxide-silver nanocomposites, microwave synthesis

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GO	Graphene Oxide
AG	Silver
Ag-NPs	Silver Nanoparticles
GO-Ag	Graphene Oxide-Silver
RGO	Reduced Graphene Oxide
UV-VIS	Ultra Violet-Visible Spectroscopy
XRD	X-ray Diffusion
FTIR	Fourier Transform Infrared
$KMnO_4$	Potassium Permanganate
$NaNO_3$	Sodium Nitrate
H_2SO_4	Sulphuric Acid
H_3PO_4	Phosphoric Acid
TiO_2	Titanium Dioxide
ZNO	Zinc Oxide
H_2O_2	Hydrogen Peroxide
$AgNO_3$	Silver nitrate
Rpm	Revolutions per Minute
HCI	Hydrochloric Acid

LIST OF SYMBOLS

α	Alpha
%	Percentage

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Graphene has recently emerged as a material with great potential for the future, and graphene oxide (GO), a highly oxidised graphene sheet, has gained popularity as a material with potential for antibacterial usage.Generally GO has a similar hexagonal carbon structure to graphene but it has functional groups such as carbonyl, carboxylic acid, hydroxyl and more.Moreover, Graphene is a single layer that makes up graphite. In other words, the graphene atoms are arranged flatly, much like billiard balls. Each layer of graphene in graphite is built up of hexagonal carbon rings that resemble several connected benzene rings, but with more carbon atoms swapping the hydrogen atoms at the edges to give the material a honeycomb-like appearance. Approximately three million layers of the attachment, each measuring one atom high, were used to fabricate a graphene sheet that was 1 mm thick (Allen et al., 2009).A silver nanocomposite is a material made of silver nanoparticles that are mixed with another material, like a polymer, a clay, or a metal. Most of the silver nanoparticles in the alloy are smaller than 100 nanometers and have special properties that make them useful in a wide range of ways. One of the best things about Ag nanocomposites is that they are better at killing germs. Silver nanoparticles have been shown to be very good at killing germs, viruses, and fungi. This makes them useful for things like healing wounds, cleaning water, and packing food. Adding Ag nanoparticles to a host material can make it stronger while keeping its antibiotic properties. Ag nanoparticles have special visual and electrical properties that make them useful for things like sensing, imaging, and making electronic devices, in addition to their ability to kill germs.(Jagiełło et al., 2020).

In the medical industry especially, silver (Ag) has been widely used for millennia. Ag nanoparticles (AgNPs) display amazing and uncommon physicochemical features and biological activity, and the convergence of nanotechnology has offered new avenues for generating pure Ag. Since then, studies using silver nanoparticles (AgNPs) have been extensively conducted, with very promising outcomes, particularly in the medical field. The majority of Malaysian industries, such as water disinfection, medicine, the textile industry, and food packaging, rely heavily on antibacterial agents. Pathogenic microorganisms such as bacteria, fungi, and viruses continue to be one of the world's greatest global health challenges, causing infectious diseases. (Szunerits, Boukherroub, Szunerits, & Boukherroub, 2018). Bacteria live in very large numbers and reproduce quickly. This is because they can actively build up drug resistance when some of the population survives treatment with antibiotics. Even though most of these diseases have gotten less common over the years thanks to commercialization and the use of standard treatments like penicillin, this has also led to more drug-resistant bacteria(Szunerits & Boukherroub, 2018).

Antibiotic-resistant bacteria are able to transfer copies of DNA coding for resistance mechanisms to closely related bacteria, resulting in the transmission of antibiotic resistance genes to future generations. Nanoparticles and nanomaterials can be used as an alternative to antibiotics because the conventional approach is less effective Jaworski et al., 2018).Thus, this study focused on UV-vis studies on reduced graphene oxide silver nanocomposite on antibacterial properties.

1.2 Problem Statement

The fabrication of graphene oxide-silver nanocomposites (GO-Ag) offers a significant obstacle within the fields of materials science and nanotechnology. Although there are numerous approaches to synthesising nanocomposites, the attainment of a controlled and reproducible synthesis process that guarantees an even distribution of silver nanoparticles across the reduced graphene oxide matrix continues to be a substantial challenge. The absence of a standardised synthesis protocol frequently results in discrepancies in the properties of nanocomposites, impeding the dependable fabrication of materials exhibiting consistent attributes. As a result, it is critical to resolve the obstacles associated with synthesis in order to fully exploit the capabilities of GO-Ag nanocomposites and facilitate their smooth incorporation into a wide array of applications, including catalysis and electronics.

Characterizing the structural and morphological attributes of graphene oxide- silver nanocomposites (GO-Ag),achieving an exhaustive characterization of nanocomposite structures is difficult due to their complexity, particularly in regards to the interaction between silver nanoparticles and the reduced graphene oxide matrix. The absence of standardised protocols for characterization frequently leads to insufficient understanding of the chemical composition and physical structure of the nanocomposite. Addressing these obstacles is of the utmost importance in order to acquire a comprehensive comprehension of the properties of the nanocomposite, which will ultimately enable the development of customised materials for particular applications.

The research may investigate how the addition of silver nanoparticles affects the absorption and reflectance spectra of the reduced graphene oxide material, how the size and concentration of the nanoparticles influence the optical properties of the nanocomposite, and what potential applications the nanocomposite may have due to its enhanced optical properties. The findings of this study could aid in the development of novel materials with enhanced optical properties for a variety of applications.

1.3 Objectives

The objectives of this research are

a)To synthesis graphene oxide - silver nanocomposite(GO-Ag)

b)To characterize the graphene oxide silver nanocomposite (GO-Ag)

c)To evaluate the optical properties of graphene oxide silver nanocomposite(GO-Ag)

1.4 Scope of Study

Graphene oxide, graphene oxide-silver (GO-Ag) nanocomposite, and graphene oxide-silver (GO-Ag) films will be produced in this experiment. Graphene oxide (GO) will be manufactured from graphite particles using a streamlined version of Hummer's method. The scope of this investigation includes the examination of multiple synthesis parameters, such as the silver nanoparticle introduction and the reduction process of graphene oxide. It is of the utmost importance to assess the impact of synthesis conditions on the reduced graphene oxide matrix in terms of silver nanoparticle morphology, size, and distribution. The objective is to develop a synthesis protocol that is optimised for the controlled production of GO-Ag nanocomposites exhibiting desirable properties.

During the characterization phase, a variety of analytical techniques will be utilised to determine the chemical, structural, and morphological properties of the graphene oxide-silver nanocomposite (GO-Ag). X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR) and Ultraviolet Visible Spectroscopy(U-vis) are some of the methodologies that will be implemented. The objective is to comprehend the dynamic relationship between silver nanoparticles and the reduced graphene oxide matrix in order to obtain a thorough characterization that offers valuable information regarding the composition and physical structure of the nanocomposite. A methodical inquiry will be undertaken to ascertain the manner in which changes in synthesis parameters influence the properties of the nanocomposite.

The optical properties component of the research endeavours to conduct a comprehensive assessment of the interaction between light and the UV-vis reduced graphene oxide-silver nanocomposite (GO-Ag). In order to thoroughly examine the absorption and transmission properties of the nanocomposite in the ultraviolet and visible regions, UV-VIS spectroscopy will be widely applied. The objective is to comprehend the effect that silver nanoparticles have on the optical characteristics of the nanocomposite. The objective of this study is to conduct an exhaustive examination of the optical characteristics of GO-Ag nanocomposites.

1.5 Significances of Study

Graphene oxide-silver (GO-Ag) film will be applied in the antibacterial application.This was demonstrated by the potent biocidal action on almost 16 different bacterial species.Additionally, this graphene oxide-silver (GO-Ag) thin film made of sodium alginate may be used in water treatment to lessen water pollution. One of the basic ingredients for human, animal, and plant existence is water. The availability of safe and clean drinking water is still a big issue in some regions of the world today, though. Every year, waterborne germs caused millions of deaths from illness.It is well established that microbial pollution of water poses a threat to human health.The procedure of water disinfection should be created to supply clean water as the answer to manage the pathogens in water. Fundamentally, there are two approaches to obtain pathogens without microbial contamination and obtain safe and clean drinking water. Numerous techniques for disinfecting water have been developed based on these concepts, including the use of chlorine, UV light, the heat-sterilized method, and size exclusion filtering. By concentrating on offering clean and sterile water for drinking, these techniques can successfully inactivate germs.

Graphene oxide is also easy to get because it can be made from graphite, which can be found in many places in nature. Also, graphene oxide has been shown to have a high bacteriostatic effect, which stops bacteria from growing and reproducing. Most other materials have a biocidal effect, which kills the bacteria at the same time. To sum up, graphene oxide is a better choice than silver compound when it comes to antibiotic agents.

Unfortunately, these practises contribute to global warming and impair the environment. Sodium alginate will be utilised to synthesise graphene oxide and silver (GO-Ag) for the production of antibacterial thin coatings.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Graphene is a two-dimensional planar sheet of sp2-hybridized carbon atoms organized in a honeycomb form, and it is categorized as atom-thick carbon with a thickness of less than one nanometer (Stankovich D. e., 2007). Graphene is a single layer of graphite. Geim and Novoselov (2004) were the first to separate graphite using mechanical exfoliation, despite the apparent impossibility of separating a single layer with a thickness of one atom. As a result, they were awarded the 2010 Nobel Prize in Physics.This 0.142 nanometer-long, single-atom-thick carbon-carbon bond consists of a two- dimensional film of sp2-bonded carbon atoms.(Banerjee, Lee, Kuila, & Kim, 2015).Due to its carbon structure, which has unique thermal, mechanical, and electrical qualities, graphene has gotten a lot of interest.(Adetayo & Runsewe, 2019).

Graphene is a single, tightly packed monolayer of carbon atoms arranged in a two-dimensional honeycomb network of sp2 hybridized carbon atoms.Graphene has recently attracted considerable attention in scientific and industrial research areas due to it's excellent properties, such as high electron mobility, excellent mechanical stiffness, extraordinary electronic transport, and high electrical conductivity.The graphene material is used in nanoelectronics,nanocomposites and energy-storage devicesFurther, graphene has recently been researched in biological applications; in addition to the physical and chemical properties already mentioned, the nanomaterial has excellent biological properties such as antibacterial,biosensing,cellular imaging, and drug delivery capabilities,as well as displaying anticancer activity.(Gurunathan ,Sangiliyandi et al.)

GO is used as a precursor for reduced GO (rGO) as it is strongly hydrophilic and generates stable and homogeneous colloidal suspensions of negatively charged GO sheets in aqueous and polar organic solvents.The subsequent deoxy-genation of GO is necessary by chemically reducing oxygen-containing groups. Maintaining the individual separation of the graphene sheets is the most important and challenging part of the rGO production process.GO reduction by chemical methods results in the formation of limited solubility or even irreversible agglomerates of rGO during preparation in water and most organic solvents (unless capping reagents are used) because of the strong π–π stacking tendency between rGO sheets.Agglomeration of rGO can be limited by attaching other molecules or polymers to the sheets. The most commonly used chemical reducing agents are hydrous hydrazine, hydrazine monohydrate, sodium borohydride, and hydrogen sulfide, which are highly toxic to living organisms and the environment.Several laboratories have developed biological reducing and stabilizing agents, such as ascorbic acid,amino acids, melatonin, glucose,humanin,microorganisms and plant extracts.(Gurunathan ,Sangiliyandi et al.)

Ag nanoparticles (AgNPs) exhibit very small sizes and large surfaces compared to the bulk metal. For several decades, AgNPs have been of considerable interest in several areas of research, including optics, electronics, magnetics, mechanics, catalysis, energy science, nanobiotechnology, and nanomedicine –

particularly as antimicrobial agents for diagnostic purposes.AgNPs also have potential applications in surface enhanced Raman scattering,catalysis,nanoscale electronics,and imaging.(Gurunathan ,Sangiliyandi et al.)

The optical properties component of the research endeavours to conduct a comprehensive assessment of the interaction between light and the UV-vis reduced graphene oxide-silver nanocomposite (GO- Ag). In order to thoroughly examine the absorption and transmission properties of the nanocomposite in the ultraviolet and visible regions, UV-VIS spectroscopy will be widely applied.

2.2 Fundamental of Graphene

Graphene was initially discovered experimentally in 2004 as a two-dimensional sheet composed of carbon atoms arranged hexagonally; it possessed a single-layer structure. Due to its two-dimensional nature and conductivity, each atom within a graphene sheet is directly exposed to its surroundings and reacts to electrostatic variations. This characteristic renders it a highly suitable material for sensing purposes, as well as for implementation in chemical vapour sensors, biomolecular sensors, and optical sensors. This carbon crystal, which is one atom thick, possesses exceptional physicochemical properties, mechanical functionality, and thermal and electrical conductivities.(Ghany N.A.A, Elsherif N.A. & Handal H.T., 2017).

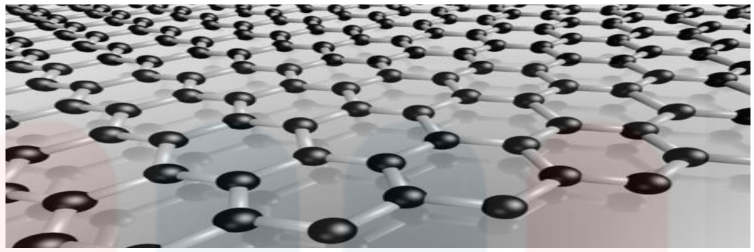

Figure 2.2: Schematic presentation of Graphene layer structure (Ghany N.A.A, Elsherif,N.A. & Handal H.T., 2017).

The valencies of carbon allotrope made graphite prevalent. Although carbon allotrope such as carbon, charcoal, and graphite also contain graphene as their fundamental structural element. A honeycomb-structured 2D lattice enhanced the mobility of charged carriers.

2.2.1 Properties of Graphene

Graphene has become one of the most hopeful nanoparticles because it has a unique mix of great qualities. There are mechanical, electrical, and heat traits among these.Graphene demonstrates plasmonic characteristics, specifically within the mid-infrared and near-infrared energy spectrum. Graphene's optical properties are influenced by the surface plasmon resonances generated by its delocalized π electrons. Gaining insight into the plasmonic characteristics of graphene is crucial in order to optimise the interaction between silver nanoparticles and graphene oxide during the synthesis process of the nanocomposite, which in turn affects its overall optical behaviour. Experts say that graphene has a breaking strength of 42 Nm-1, a Young's modulus of 1.0 TPa, and an intrinsic tensile strength of 130.5 GPa(Smith et al., 2019). It was thought that graphene oxide could be used as a mechanical support to make a high-performance composite material that has a high stiffness, high strength, low thickness, and is light (Cheng-An et al., 2017).Smith et al. (2019) say that adding graphene to plastics has made them much better at conducting electricity.A pure graphene sheet makes it hard to make a nanocomposite because it is chemically steady and inactive, even though it has many great qualities.

2.3 Fundamental of Graphene Oxide (GO)

Graphene oxide (GO) is created by prolonged oxidation of graphite or graphene using three essential principles: Brodie, Staudaumaier, and Hummers. This theory is based on the oxidation of graphite or graphene in the presence of a strong acid and an oxidizing agent.Furthermore, graphene oxide (GO) is one of the most common graphene instances (Merritt, Wan, Shollock, & Patole).GO is one of the most important graphene-based substances (Yan et al., 2014).A single monomolecular layer of graphite with oxygen-containing functional groups including epoxy, hydroxyl, carbonyl, and carboxyl makes up the distinctive substance known as GO. Utilizing these characteristics makes it easier to get excellent graphene oxide dispersion in water and other solvents, which makes it simpler to create polymer nanocomposites and scale up graphene oxide synthesis.The use of GO in electrically conductive materials is limited by the covalent oxygen functional groups in GO, which generate structural defects that have a major impact on properties such as electrical conductivity.Furthermore, Figure 2.3 demonstrated the process of chemically synthesising graphene from graphite.

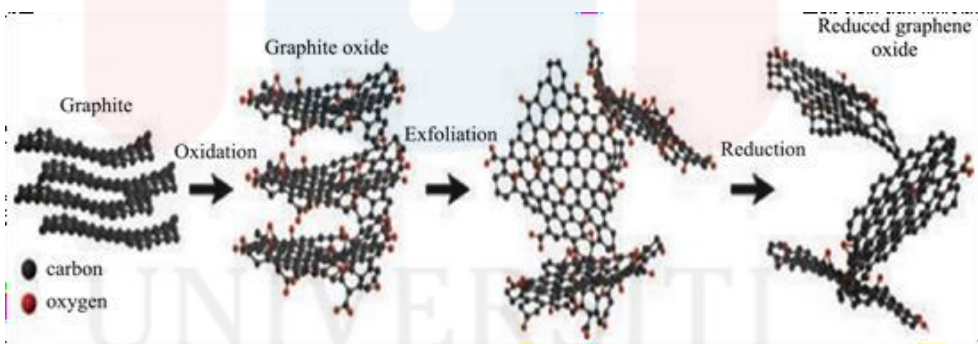

Figure 2.3 Synthesis route of graphene from graphite

2.3.1 Properties of Graphene oxide

The relative simplicity with which graphene oxide can be produced in large quantities can be used to compare its qualities. Graphene oxide continues to be utilised in composites, biosensors, electronics, energy storage, healthcare, and other applications.Yang et al. (2014) conducted extensive research on the synthesis of GO-MNPs from noble metal nanoparticles, specifically gold and silver, for future applications in catalysis, electrochemistry, microbicide, and surface-enhanced Raman scattering (SERS).A substance with a high electrical resistance is graphene oxide. This is due to the fact that the process of disrupting the sp2 bonding orbitals of 10 graphene and adding numerous surface groups inhibits its electrical conductivity, rendering GO electrically resistive (1.64 104 m) during the production of GO (Smith et al., 2019).Smith et al. (2019) also suggested that the high resistance of GO could be reduced by converting GO to reduced graphene oxide, rGO, via a reduction process. This strategy will resolve the properties of the network by reconstructing it.

2.3.2 Synthesis of Graphene Oxide

Throughout the years, numerous researchers have investigated numerous methods for manufacturing graphene oxide. Hummer's process, which entails oxidising graphite particles, is the most common procedure for producing GO (Jaworski et al., 2018).The most well-known method for manufacturing GO is Hummer's technique (KMnO4, NaNO3, H2SO4). However, according to Marcano et al. (2010), removing NaNO3, increasing the amount of KMnO4, and conducting the reaction in a 9:1 mixture of H2SO4 and H3PO4 will increase the oxidation efficacy. According to reports, this technique produces more hydrophilic oxidised graphene material than Hummer's technique.

2.4 Graphene Oxide silver (GOAg) nanocomposite

Silver has been used for a very long time because it kills germs. Humans say that the Persians used metal containers to store water, especially during wars, to make sure the water stayed clean. The ancient Phoenicians, Greeks, Romans, and Egyptians also used silver to keep food and water fresh, according to some records. Silver has been used for many things, but it is most often used to kill germs. Now that silver nanoparticles are better, it can be used in the best way possible.About 120 years ago, in 1889, McLea wrote that he had made a citrate-stabilized silver colloidal material. This was the first time that nano-silver was talked about. McLea made a silver colloid with a width of 7 to 9 nm. This sparked interest in this material in the fields of physics, chemistry, biology, and materials science.However, scientists have found it hard to make silver without it clumping together and falling apart. Silver nanoparticles can easily stick together and form clumps in water, which could make it less useful and less effective at killing germs. Abd-Elaal, Tawfik, et al. (2015) and Charistoudi, Kallitsakis, et al. (2017) say that using surfactants and reducing agents could reduce agglomeration. However, it should be noted that using surfactants and reducing agents is still dangerous for humans and the environment.Also, using a detergent could stop the surface from oxidising, which would slow the rate of Ag+ions and make them less dangerous to bacteria.The functional group on GO works as a starting point for AgNPs. This change not only improves the surface area of AgNPs, but it could also improve their ability to stick together, connect with other surfaces, and stay stable (Agnihotri, Mukherji et al., 2014).

The association between the functional group and the Ag+ in the AgNPs and the GO sheets could keep the AgNPs from sticking together. Since the clumping is getting less, more Ag+ ions could get out, which could make it more dangerous to bacteria. There are many ways to make GOAg, such as chemical reduction, electrochemical reduction, hydrothermal/solvothermal methods, and the template-assisted approach. In 2014, one study successfully made the GOAg nanocomposite and used sodium citrate as a binding agent to boost its antibiotic action. It was said that the nanocomposite formed had good dispersion in water and had circular forms with an average size of less than 10 nm.

2.5 Microwave Synthesis

Silver graphene oxide nanocomposites was fabricated using rapid and microwave irradiation synthesis method (Chook S. et al., 2012). The solution of silver graphene oxide was moderately exposure to the microwaves irradiation for 30 second, 40 second, 50 second, 60 second and 70 second. The effect of microwave irradiation will cause narrow size distribution.

The morphology of the cross-sectional, plane, phase characteristic and structure of graphene can be observed by using other techniques which are X-ray diffraction, Scanning Electron Microscopy, UV-Vis and Fourier Transform Infrared Spectroscopy.

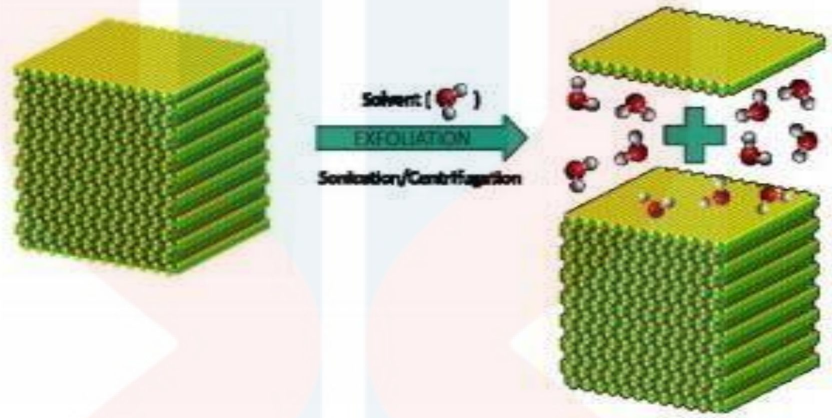

Figure 2.5 Schematic diagram showing the exfoliation of graphite to produce graphenesheets (Ghany N.A.A, Elsherif,N.A. & Handal H.T., 2017).

2.6 Processing Method of Graphene Oxide-Silver Nanocomposite

Yang et al. (2014) used in situ fabrication to reduce graphene oxide, which is said to be easy and systematic. Many of the reducing and stabilizing chemicals employed in this manufacture, such as hydrazine and sodium borohydrate, are poisonous, and the procedure is usually difficult. Hydrazine hydrate was employed as a reducing agent to break down graphene oxide because other powerful reducing agents do not function with water and because it aids in the formation of sheets and films that resemble graphite. Microwave treatment has been adopted to replace in situ manufacture to alleviate the issue of the toxic stabilising agent. This is because it utilizes less energy than standard heating techniques.(Chook et al., 2012).

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

Graphite powder is one of the main things that used in this thesis. The material used 3g graphite powder. Others materials used were 320 ml sulfuric acid (H_2SO_4) 9%, 80 ml phosphoric acid (H_3PO_4), 18g potassium permanganate ($KMnO_4$), 27mL hydrogen peroxide (H_2O_2) 37%, 10 mL 10 milliMolar (mM) silver nitrate ($AgNO_3$), 1M hydrochloric acid (HCI), ammonia solution (25%), 70% of ethanol

3.2 Methodology

3.2.1 Preparation of graphene oxide

Graphite powder and a simpler version of Hummer's method will be used to make graphene oxide. First, 3 g of graphite powder is mixed with 320 mL of sulfuric acid H_2SO_4 ,80 mL of phosphoric acid H_3PO_4 , and 18 g of potassium permanganate $KMnO_4$ to make the mixture. To get the most graphite to oxidise, the mixture will be mixed slowly for 3 days with a magnetic mixer. The mixture will then be added to the H_2SO_4 to stop the oxidation process once the solution's colour has changed from dark purple-green to dark brown. The mixture will be split evenly among six centrifuge bottles that will be cleaned. The GO solution will be washed three times with 1 M of HCI and pure water at a speed of 10,000 rpm, until a pH of 5 is reached.

Figure 3.2.1: Preparation of Graphene Oxide

3.2.2 Synthesis of G0-Ag nanocomposite

Prepare 100 ml 50 mM $AgNO_3$ nitrate.Then,Prepare 10 ml of 20 mM $AgNO_3$.Around 80 mL ammonia (25 wt%) was slowly added into 10 mL of silver nitrate solution (20 mM). The mixture was vigorously stirred until a clear solution was observed indicating the formation of complex [Ag(NH_3)20H].The solution was then mixed with 10 ml of 0.5 mg/mL of aqueous GO solution.Then, the reaction mixture was exposed to the microwave irradiation for 30s,40s,50s,60s,70s.The GO-Ag mix will then go through the microwave synthesis will be ultra-sonicated. When the Ag-GO solution nanocomposite makes a solution with a yellowiprocess and then be cooled to room temperature. About 180°C (356°F) is how hot the microwave is. This heating method will be used as a catalyst to help Ag nanoparticles stick to the surface of GO.

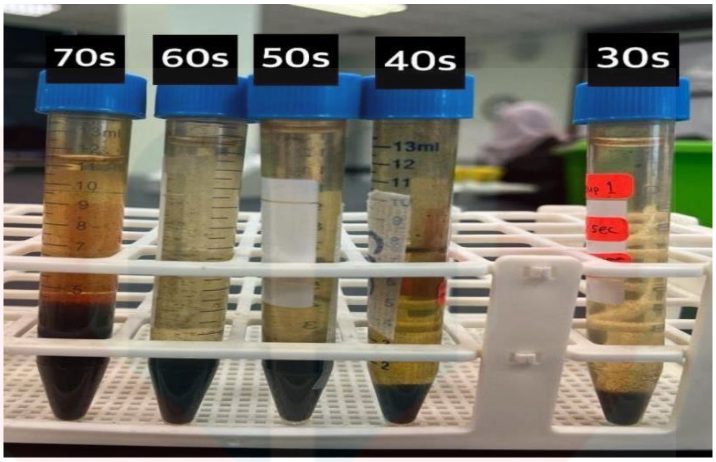

Figure 3.2.2:Mixture of Ag-GO after irradiate by using the microwave for 30s,40s,50s,60s and 70 seconds.

3.3 Characterization

To characterize the crystal structure, morphology, elemental composition, physical and chemical properties, and applications, various methods of characterization will be applied to the sample for various purposes.

3.3.1 UV-Vis Spectroscopy

The study of several techniques that may be used to produce spectra is known as spectroscopy. UV-vis spectroscopy functions on the fundamental principle that electrons endure electronic transitions within atoms or molecules when a sample is exposed to ultraviolet or visible light. The transitions in question are associated with the absorption of light at particular wavelengths, which gives rise to distinctive absorption spectra. UV-vis spectroscopy permits the identification and characterization of electronic transitions linked to the existence of silver nanoparticles and graphene oxide within the framework of nanocomposites such as GO-Ag.

The interpretation of the obtained spectrum may be used to investigate molecular structures, assess the makeup of a substance, evaluate elements and chemical compounds, and so on. The absorbance of GO-Au nanocomposites will be measured by UV-Vis scanning from 190 to 900 nm. It will offer an absorbance value based on the electronic and energetic states of the material. The samples will be distributed in quartz cuvettes and analyzed using a UV-Vis analyzer from Thermo Scientific. By examining the absorption data from UV-VIs spectroscopy, it is feasible to determine if silver nanoparticles and plasmonic resonance are being produced. A UV-Vis Spectrophotometer may also be a tool or a method for measuring the state of aggregation as well as the size promptly, non-invasively, and in real time.Critical to the overall efficacy of GO-Ag, the interaction between silver nanoparticles and graphene oxide can be deduced from the spectroscopic data.Underlying the premise of UV-vis spectroscopy is that electrons endure electronic transitions within atoms or molecules when a sample is exposed to ultraviolet or visible light. These transitions give rise to distinct absorption spectra, which are determined by the absorption of light at particular wavelengths. UV-vis spectroscopy is a valuable technique for ascertaining and quantifying electronic transitions that are linked to the existence of silver nanoparticles and graphene oxide in nanocomposites such as GO-Ag.

Figure 3.3.1: UV-Vis Spectrophotometer

3.3.2 Fourier Transform Infrared Spectrometer (FTIR)

A very popular technique, Fourier transform infrared spectroscopy (FTIR), uses an infrared light beam to identify functional groups in materials (gas, liquid, and solid). A infrared spectroscopy, which analyses the amount of infrared radiation that is absorbed by each bond in a molecule, produces

a spectrum that is typically described as a percentage of transmittance versus wave number (cm1). (Shahid Ali Khan et al., 2018).It will also validate that chemicals were removed following the bleaching procedure. Following microwave synthesis, the GO-Ag nanocomposite samples will be examined using an iZ10 FT-IR Spectrometer, with wave numbers ranging from 400 cm-1 to 4000 cm-
1. The sample's liquid form will be used.

3.3.3 X Ray Diffraction (XRD)

One of the best techniques for identifying unresolved crystalline phases is X-ray diffraction. To determine a substance's crystalline structure, a technique known as X-ray diffraction analysis, or XRD for short, is employed in the field of materials science. Crystalline compounds are identified by comparing the positions and intensities of diffraction peaks to a database. The phenomenon of incoming X-ray beams interfering with one another as they leave a crystal due to the atomic planes of the crystal is known as X-ray diffraction. The analytical technique of XRD is used to recognize crystalline materials and to give data in unit cell dimension.As a result, when electromagnetic waves hit a regular array of scatters, X-ray diffraction patterns are created. The collecting and upkeep of the crystal structures of a wide range of materials are handled by the International Centre for Diffraction Data (ICDD). On the computers that run the tools that are used to identify the unidentified chemicals and their phases, the data files were subscribed to and then saved. The standard patterns that were saved in the database were contrasted with the diffracted patterns of the unknown materials.The required region's size and shape, as well as the average between layers and rows, will all be calculated by XRD. GO-Ag's crystalline structure will be studied using an XRD equipment from Bruker AXS Germany that was scanned from 10° to 80°. The sample will be freeze-dried to create an XRD analysis powder.

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CHAPTER 4

RESULTS AND DISCUSSION

4.1 Ultra Violet Visible Spectroscopy of Ag-GO Nanocomposite

The absorbance value suggests the electronic and energy states of the sample based on the UV- Vis function. It was utilised to observe the formation of silver nanocomposites on Ag-GO nanocomposite in this experiment. Two peaks will be discernible in the UV-Vis spectrum, representing the peaks of GO and Ag-GO, respectively. As shown in Figure 4.1, GO will exhibit a spectrum signal at 232 nanometers and a shoulder at 300 nanometers. The π–π* transitions of aromatic C–C bonds are defined at 232 nm, whereas the n–π* transitions of C=O bonds are the basis for GO at 300 nm. A novel peak appeared at 420nm subsequent to the microwave synthesis of the silver graphene oxide nanocomposite solution, indicative of the development of Ag NPs. Surface Plasmon Resonance (SPR) absorption band: Ag nanocomposites exhibit an absorption band at approximately 420nm in wavelength. During microwave irradiation, the intensity of the SPR band at 30s, 40s, 50s, 60s, and 70 seconds of the Ag-GO nanocomposite varied, resulting in absorbance peaks between 400 nm and 416 nm that were distinct. Furthermore, an analysis of Figure 4.1(30s,40s,50s,60s,70s) reveals that the absorbance peak at 230 nm no longer represents the reduction of GO and Ag+ to Ag nanocomposites.

Figure 4.1 illustrates that the broad and narrow peak observed at 30s,40s and 50 seconds of the microwave synthesis of Ag-GO nanocomposite differs from the sharp and narrow peaks observed at 60s and 70s, which result from an increase in the microwave synthesis time, respectively. The SPR peaks of Ag-GO nanocomposites subjected to irradiation for 30s ,40s and 50 seconds shifted marginally to the right (approximately 420 nanometers) and exhibited a longer wavelength at higher AgNO3 concentrations. The incorporation of 25% ammonia enhanced and facilitated the development of AgNPs on the graphene layer (Gurunathan ,Sangiliyandi et al & Ciptasari et al., 2022)

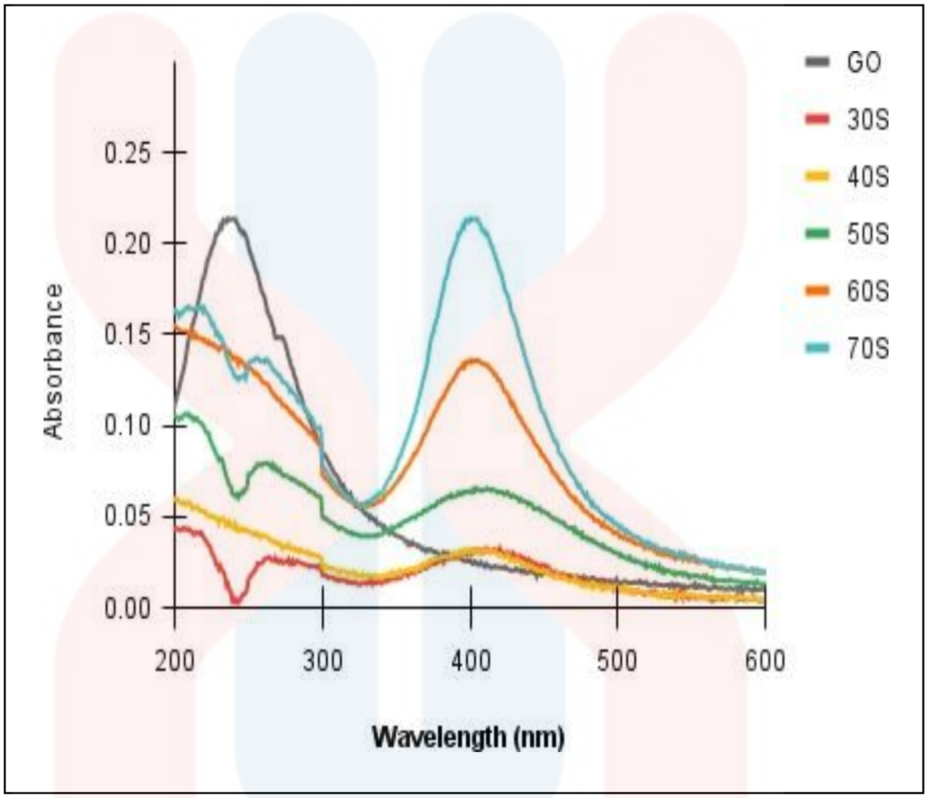

Figure 4.1: UV-visible absorption spectra of GO and Ag-GO nanocomposite for 30s ,40s , 50s, 60s and 70s

4.2 FTIR studies of Ag-GO Nanaocomposite

The interactions between GO and Ag was investigated by using Fourier Transform Infrared (FTIR) spectra.Figure 4.2 illustrates the spectra of Ag-GO nanocomposite and GO.The broad and extensive spectra indicate that GO and Ag-GO both comprise a significant quantity of hydroxyl groups.From FTIR spectra of GO, the broad and intense peak positioned at 3193 cm^{-1} which resulted to OH groups. The adsorption band at approximately 1620 cm^{-1} corresponds to the C=O bonding by the aromatic rings of the GO carbon skeleton, structure which also identified as intra-molecular hydrogen bonds.The GO surface exhibited an abundance of oxygenous and hydroxyl groups, which facilitated modification by plasmonic nanoparticles, specifically Ag nanoparticles in this experiment.

In Figure 4.2, FTIR spectrum of Ag-GO nanocomposites contain 5 broad and intensepeak which represent of microwave synthesize time. At Figure 4.2(b) the peaks at 3285.15 cm^{-1}, 4.2(c) the peaks located at 3267.20 cm^{-1}, 4.2(d) at 3265.91cm^{-1}, 4.2(e) at 3262.43 cm^{-1} and 4.2(f) positioned at 3257.30 cm^{-1} which resulted to OH groups. While the adsorption band at 1636.33 cm^{-1} (Figure 4.2b), 1636.26 cm^{-1} (Figure 4.2c), 1636.24 cm^{-1} (Figure 4.2d),1636.13 cm^{-1} (Figure 4.2e), 1636.00 cm^{-1} (Figure 4.2f), corresponds to the C=O bonding by the aromatic rings of the GO carbon skeleton. This peak of functional group proves that graphite are successfully oxidize at GO.(Gurunathan,Sangiliyandi et al & Ciptasari et al., 2022)

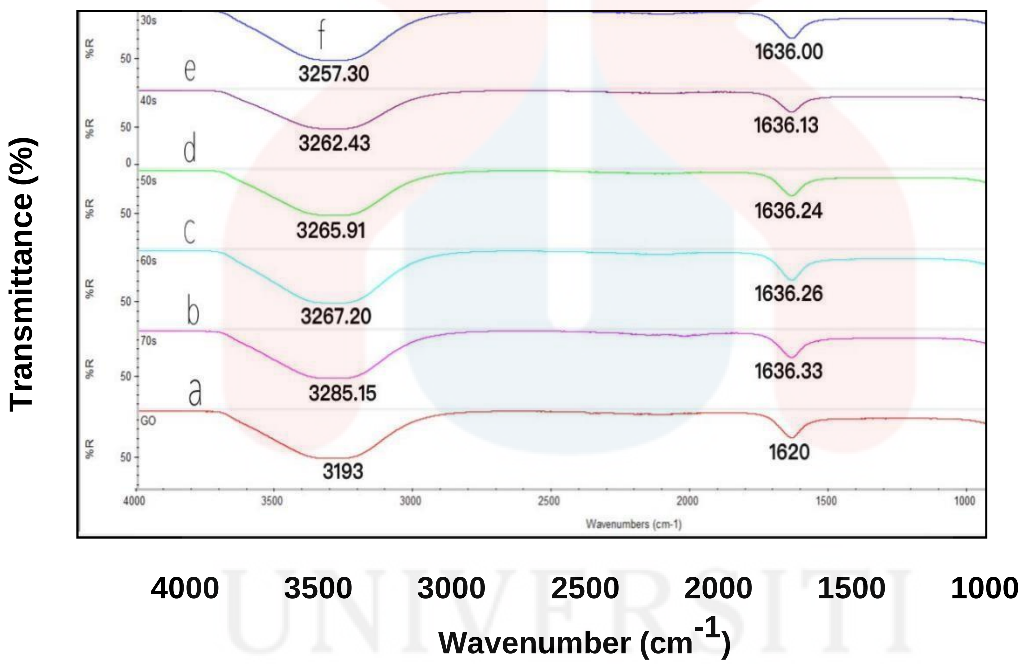

Figure 4.2: FTIR patterns of GO (a) and Ag-GO that synthesized by microwave at 30 s (f), 40 s (e), 50 s (d), 60 s (c) and 70 second (b).

4.3 XRD studies of Ag-GO Nanocomposite

XRD was utilised in this investigation to examine the structure of Ag-GO and GO nanocomposites. The presence of Ag nanoparticles is verified in Figure 4.3 through the identification of diffraction patterns of the Ag crystal structure that correspond to the standard X-Ray Diffraction (XRD) pattern.XRD structural analysis of GO nanocomposite shown in Figure 4.3 which the strong diffraction peak at 2 Theta (degree) characterize at value 10.0˚ resembles to the (00 1) which represent of crystalline nature of GO. After microwave synthesis of GO and Ag nanoparticles at 40s, 50s, 60s and 70 seconds, the formation of Ag-GO nanocomposite confirmed by the presence Ag crystal structure diffraction pattern that match with standard XRD pattern. The XRD patterns obtained from the black powders demonstrated the presenceof silver in the products, and the peaks can be indexed to the diffraction peaks at 38.1˚, 44.1˚,64.2˚ and 77.2˚ which correspond to the crystallographic planes (1 1 1), (2 0 0), (2 2 0) and (3 1 1) which the crystal planes was face-centered cubic silver structure.The high intense diffraction peak observed at 38.1◦, corresponding to the crystalline Ag nanoparticles, approves that the nanoparticles composed of pure crystalline Ag. In (Figure 4.2) shown that Ag nanoparticles peaks at (1 1 1) ,(2 0 0) and (220) crystallographicplanes outstanding for all the $AgNO_3$ concentration used. Although, there were one crystallographic planes on (3 1 1) of the Ag nanoparticles cannot recognized because of low concentration. From the Figure 4.2 shown that the regular layer structure of GO totally demolished by $AgNO_3$ solution. The intensity of Ag diffraction peaks increasing in line with the concentration of the silver and graphene oxide during the microwave synthesis.(Gurunathan ,Sangiliyandi et al & Ciptasari et al., 2022)

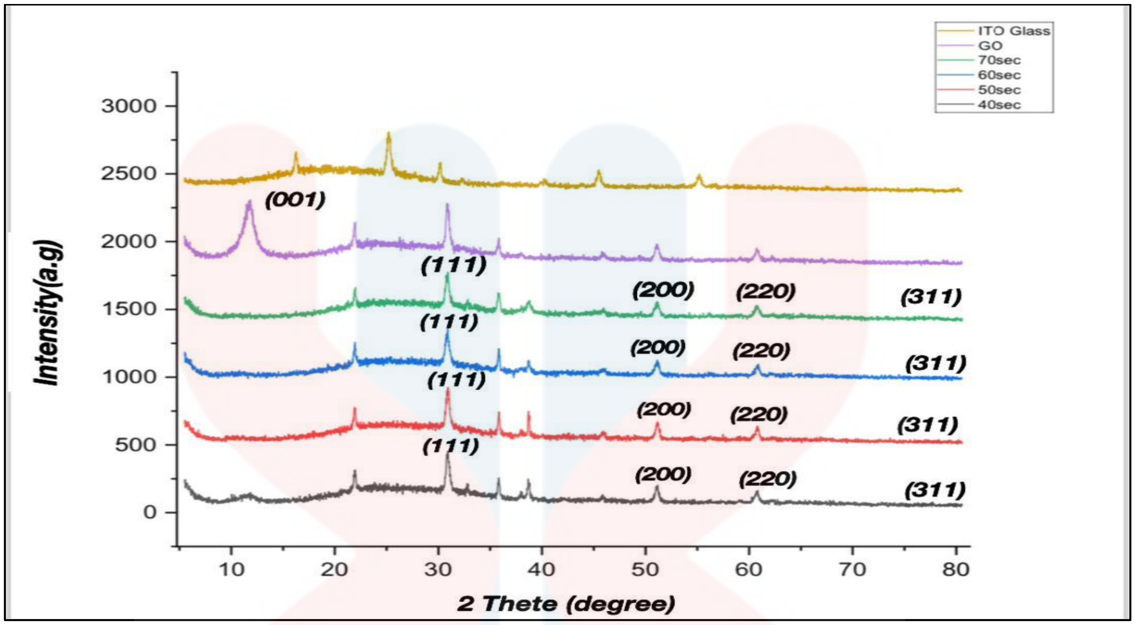

Figure 4.3: XRD pattern of GO and Ag-GO nanocomposite synthesis by microwave at 40 second , 50 second, 60 second and 70 second

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# CHAPTER 5

## CONCLUSIONS AND RECOMMENDATIONS

### 5.1 Conclusions

In summary, I successfully achieve objective that is To synthesis graphene oxide - silver nanocomposite(GO-Ag), To characterize the graphene oxide silver nanocomposite (GO-Ag) and To evaluate the optical properties of graphene oxide silver nanocomposite(GO-Ag).This works was successfully synthesized Ag-GO nanocomposite by using the microwave synthesis method.The UV-vis reduced graphene oxide-silver nanocomposite (GO-Ag) has been extensively characterised by means of UV-vis spectroscopy, FTIR, and XRD, yielding significant knowledge regarding its optical, morphological, and structural attributes. The findings derived from each characterization methodology enhance the overall comprehension of the nanocomposite that was synthesised.The UV-vis spectroscopy analysis reveals that the GO-Ag nanocomposite was effectively synthesised, as evidenced by the presence of distinct peaks at 232 nm and 300 nm that correspond to graphene oxide (GO) and a new peak at 420 nm that signifies the formation of silver nanoparticles (Ag NPs), respectively. Approximately 420 nanometers in wavelength, the Surface Plasmon Resonance (SPR) absorption band is detected, providing additional confirmation of the existence of Ag nanocomposites. Variations in the intensity of the SPR band during distinct microwave synthesis times indicate that the nanocomposite's composition undergoes dynamic
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changes.FTIR Analysis are the interactions between Ag and GO in the nanocomposite were revealed by FTIR analysis. Both Ag-GO and GO exhibit widespread peaks in their FTIR spectra, which are indicative of hydroxyl groups. The discernible peaks observed during various microwave synthesis times offer valuable information regarding the modifications in functional groups, specifically the C=O and OH bonding of the aromatic rings. The affirmation of the effective oxidation of graphite to GO is accompanied by discernible variations in the FTIR spectra, which indicate the presence of Ag nanoparticles.XRD Structural Analysis are the structural modifications introduced by the microwave synthesis of Ag nanoparticles and GO are validated by the XRD analysis. Crystalline character of GO is confirmed by the prominent diffraction peak at 10.0˚ in its XRD pattern. At various synthesis periods, the subsequent XRD patterns of Ag-GO nanocomposites display diffraction peaks that correspond to the standard XRD pattern observed in silver crystals. Specific crystallographic planes (1 1 1), (2 0 0), (2 2 0), and (3 1 1) exhibit indexed diffraction peaks that validate the face-centered cubic structure of silver nanoparticles.Overall, By integrating UV-vis spectroscopy, FTIR, and XRD analyses, a comprehensive comprehension of the GO-Ag nanocomposite's structural properties and effective synthesis can be attained. The findings as a whole illustrate the integration of silver nanoparticles into the graphene oxide framework, suggesting a wide range of potential uses in disciplines including catalysis, sensing, and optoelectronics. The effects of microwave synthesis time on the properties of the nanocomposite are highlighted by the dynamic alterations in absorption bands, functional groups, and crystallographic planes. This investigation substantially enhances the body of knowledge regarding UV-vis reduced graphene oxide in silver nanocomposites, thereby creating opportunities for additional scrutiny and implementation in the field of advancedmaterials science.

5.2 Recommendations

There were several suggestions for future research pertaining to this study, should it be continued.In future research endeavors, it is recommended to expand the characterization of the UV-vis reduced graphene oxide-silver nanocomposite (GO-Ag) by delving into various aspects. Firstly, a deeper understanding of the chemical composition can be achieved by employing techniques like X-ray photoelectron spectroscopy (XPS) to analyze the surface chemistry and oxidation state of GO-Ag, shedding light on the interaction between graphene oxide (GO) and silver nanoparticles. Morphological analysis can be enhanced through high-resolution transmission electron microscopy (TEM) and atomic force microscopy (AFM) to study the spatial distribution, size, and shape of Ag nanoparticles on the GO surface. Additionally, exploring the electrical and thermal properties using techniques such as four-probe resistance measurements, impedance spectroscopy, thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC) can provide a more comprehensive understanding of GO-Ag.

To tailor the optical properties of GO-Ag, future research can focus on achieving controllable Ag nanoparticle size and shape through the optimization of synthesis parameters. Doping and functionalization of GO with different elements or organic molecules can be explored to fine-tune the absorption and emission properties. The creation of multilayer structures with alternating layers of GO and Ag nanoparticles could lead to enhanced optical effects.

In terms of applications, the potential of GO-Ag in various fields can be further explored. Sensors utilizing the unique optical properties of GO-Ag for sensitive detection of specific molecules or ions could be investigated. The photocatalytic activity of GO-Ag for applications in water purification, pollutant degradation, or hydrogen generation can be evaluated. Optoelectronic devices such as solar cells, light-emitting diodes (LEDs), or photodetectors may benefit from the incorporation of GO-Ag. Furthermore, the potential biomedical applications of GO-Ag, including bioimaging, drug delivery, and antibacterial coatings with minimal cytotoxicity, warrant thorough exploration.

In addition to these scientific aspects, considerations for scalability and cost-effectiveness in synthesis methods for large-scale production, an assessment of environmental impacts, and collaboration with researchers from diverse disciplines could enrich the holistic understanding and applications of GO-Ag. These future research directions aim to contribute to the advancement of knowledge and the potential utilization of GO-Ag in various technological domains.

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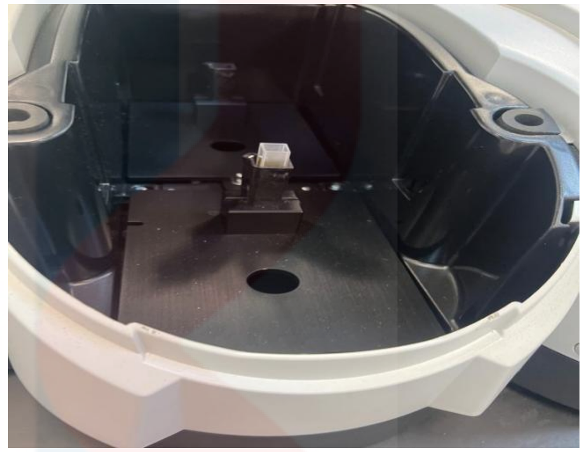
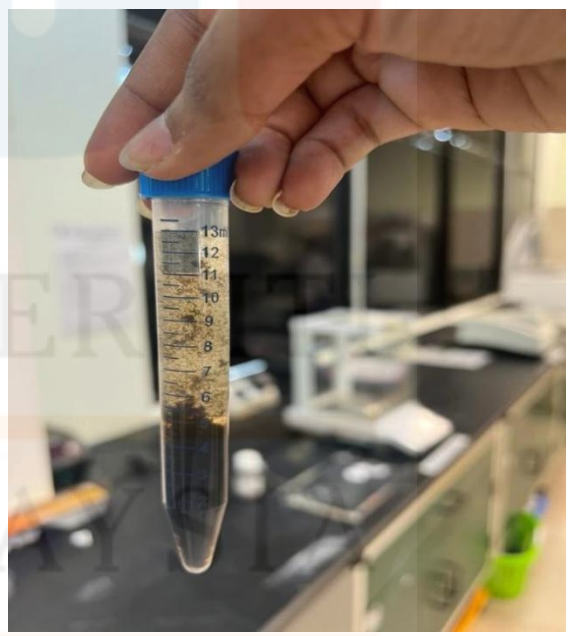
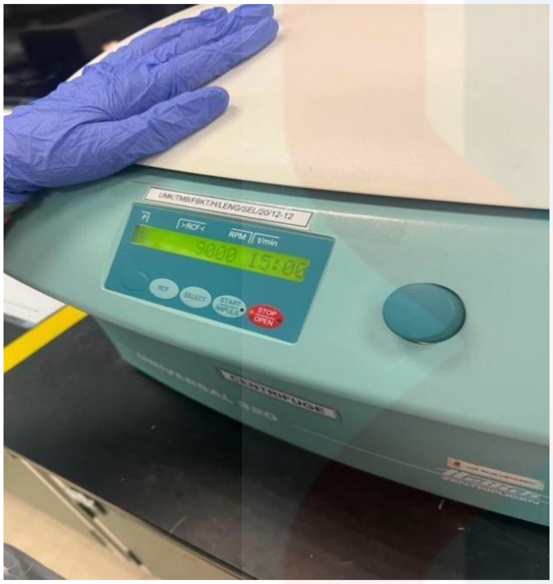
APPENDIX A





APPENDIX B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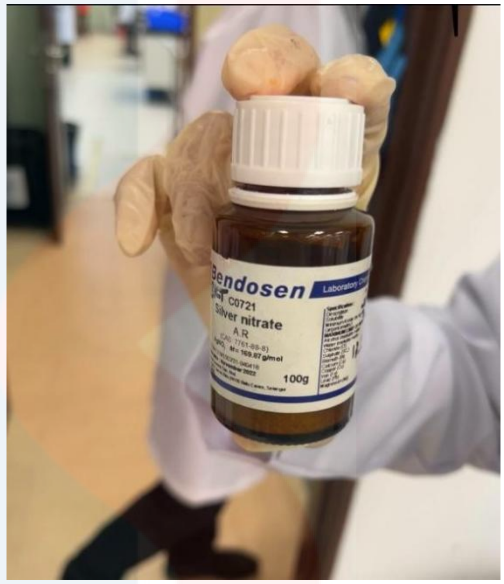
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