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**XRD studies on reduced Graphene Oxide-Aurum  
Nanocomposite**

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## DECLARATION

I declare that this thesis entitled “XRD studies on reduced GO-Au Nanocomposite” is the result of my own research except as cited in the references.

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## **XRD studies on reduced Graphene Oxide-Aurum Nanocomposite**

### **ABSTRACT**

The utilization of reduced graphene oxide (rGO) as the foundational material in order to stabilize gold nanoparticles (AuNPs) is presented as a novel approach to the production of hybrid nanocomposites. It is the primary purpose to incorporate low-energy gold (Au) ions, which will result in the creation of reactive sites on rGO. This will, in turn, make it easier for AuNPs to nucleate and intercalate within a few layers of rGO. An unique characterization of these nanocomposites is accomplished by the utilization of X-ray diffraction (XRD), optical microscopy (OM), ultraviolet-visible spectroscopy (UV-Vis), and Fourier-transform infrared (FTIR) in the process. Crystallographic changes may be visualized using OM, morphological changes can be visualized using XRD, optical characteristics can be determined using UV-Vis spectroscopy, and chemical modifications can be discovered using FTIR. Significantly, the findings provide evidence that the implantation of Au ions was carried out successfully, hence shedding light on surface roughening and altered chemical states in the rGO nanostructure. The significance of our method in achieving targeted alterations within the nanocomposite structure is highlighted by this discovery, which demonstrates the potential of our technique for the development of revolutionary material strategies. We have a better grasp of the one-of-a-kind characteristics of rGO-AuNP nanocomposites as a result of the complete characterization, which used techniques such as XRD, OM, UV-Vis, and FTIR. The contributions made by this author provide useful insights that could be used to a wide variety of study and technology fields.

**Keywords:** Aurum, graphene-oxide, nanoparticle, nanocomposite

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## XRD studies on reduced Graphene Oxide-Aurum Nanocomposite

### ABSTRAK

Penggunaan graphene oxide (rGO) yang dikurangkan sebagai bahan asas untuk menstabilkan nanopartikel emas (AuNPs) dipersembahkan sebagai pendekatan baru untuk pengeluaran nanokomposit hibrid. Ia adalah tujuan utama untuk menggabungkan ion emas (Au) tenaga rendah, yang akan menghasilkan penciptaan tapak reaktif pada rGO. Ini seterusnya akan memudahkan AuNPs untuk nukleus dan interkalasi dalam beberapa lapisan rGO. Pencirian unik nanokomposit ini dicapai dengan penggunaan pembelauan sinar-X (XRD), mikroskop optik (OM), spektroskopi boleh dilihat ultraungu (UV-Vis), dan inframerah transformasi Fourier (FTIR) dalam proses tersebut. Perubahan kristalografi boleh divisualisasikan menggunakan OM, perubahan morfologi boleh divisualisasikan menggunakan XRD, ciri optik boleh ditentukan menggunakan spektroskopi UV-Vis, dan pengubahsuaian kimia boleh ditemui menggunakan FTIR. Secara ketara, penemuan memberikan bukti bahawa implantasi ion Au telah dijalankan dengan jayanya, justeru memberi penerangan tentang kekasaran permukaan dan mengubah keadaan kimia dalam struktur nano rGO. Kepentingan kaedah kami dalam mencapai perubahan yang disasarkan dalam struktur nanokomposit diserlahkan oleh penemuan ini, yang menunjukkan potensi teknik kami untuk pembangunan strategi bahan revolusioner. Kami mempunyai pemahaman yang lebih baik tentang ciri-ciri satu-satu-jenis nanokomposit rGO-AuNP hasil daripada pencirian lengkap, yang menggunakan teknik seperti XRD, OM, UV-Vis dan FTIR. Sumbangan yang dibuat oleh penulis ini memberikan pandangan berguna yang boleh digunakan untuk pelbagai bidang kajian dan teknologi.

Kata kunci: Aurum, graphene-oxide, nanopartikel, nanokomposit

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**LIST OF ABBREVIATIONS (optional)**

GO	Graphene Oxide
rGO	Reduced Graphene Oxide
AuNPs	Aurum Nanoparticles
Au	Aurum
XRD	X-Ray Diffraction
OM	Optical Microscopy
UV-Vis	Ultraviolet Visible
FTIR	Fourier Transform Infrared
HAuIC <sub>4</sub>	Chloroauric acid
cm <sup>-1</sup>	Reciprocal centimetres
nm	nanometre
mL	millimetre
Rpm	Revolutions Per Minute

## LIST OF SYMBOLS (optional)

°

Degree sign

=

Equal sign

$\theta$

Theta

@

At sign

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of Study

In recent years, graphene itself as well as other materials based on graphene have emerged as crucial components of composite science and technology (Strankowski et al., 2016). Not only is graphene the sole material that has these one-of-a-kind qualities, but descendant molecules like graphene oxide, reduced graphene oxide, customized graphene, and so on do as well (Strankowski et al., 2016). To produce graphene oxide (GO), graphite may be oxidized, which is recognised as one of the most significant materials from a structural standpoint (Strankowski et al., 2016). One way to look at it is as a single monomolecular layer of graphite that contains a variety of oxygen-containing functional groups, including epoxide, carbonyl, carboxyl, and hydroxyl groups (Haldorai et al., 2014). Graphene oxide (GO) has high conductivity and is used in sensors, anticancer characteristics, electronics, biomedical antimicrobial coatings, photocatalytic activity, water purification, solar desalination, and drug delivery (Kumari et al., 2020).

The removal of oxide groups results in the creation of vacancies and structural flaws, which alters the compatibility of polymer chains with (rGO) (Strankowski et al., 2016). Due to the presence of extra nanosized adhesion areas on the surface and edges of reduced graphene oxide flakes, reduced graphene oxide (rGO) is a much more effective filler in composites that are otherwise comparable to GO (Strankowski et al., 2016).

Ascorbic acid reduction of graphene oxide to reduced graphene oxide (rGO) is another low-cost and eco-friendly option. While environmentally friendly, this technique has not been tested with varying inputs. Various factors have been used in the production of graphene oxide in this study. Reduced graphene oxide (rGO) and its nanocomplex have been the subject of research for potential uses in antimicrobial therapy, wound healing, medication delivery, and cancer prevention (Ahamed et al., 2021).

A nanocomposite material that contains nanoparticles of gold (Au) is referred to as having the "aurum nanocomposite" designation. Because of the one-of-a-kind optical, electrical, and catalytic capabilities that gold nanoparticles possess, they have found widespread use in a variety of sectors (Han et al., 2012). When they are made into a nanocomposite with other materials, they may display functionalities that are either improved or newly discovered. The particular qualities and uses of an aurum nanocomposite would be contingent on the other materials used and the technique of synthesis that was utilised. It is possible to create a nanocomposite material by dispersing gold nanoparticles across a matrix of polymer molecules. These materials have the potential to display fascinating optical features, such as plasmonic behaviour. This is a phenomenon in which the gold nanoparticles' localised surface plasmon resonance interacts with light. Nanocomposites made of aurum and polymers have found use in a variety of fields, including optoelectronics, sensors, and biomedical devices

In order to create nanocomposites, gold nanoparticles may be mixed with materials consisting of metal oxides, such as titanium dioxide (TiO<sub>2</sub>) or iron oxide (Fe<sub>3</sub>O<sub>4</sub>). When compared to the qualities of the separate components, these materials could have enhanced photocatalytic or magnetic capabilities. Nanocomposites made of aurum and

metal oxides have found use in a variety of fields, including photocatalysis, solar cells, and magnetic resonance imaging (MRI).

In a nutshell, a nanocomposite material is said to be an Aurum nanocomposite if it contains gold nanoparticles in its composition. It is possible to produce one-of-a-kind characteristics and functions by mixing gold nanoparticles with other materials, which has led to a broad variety of applications in a variety of sectors, including biomedicine, electronics, and optics, as well as catalysis.

## **1.2 Problem Statement**

While graphene oxide-gold (GO-Au) nanocomposites hold promise for various applications, including biomedicine, electronics, and optics, their crystal structure and phase composition remain inadequately characterized. Existing X-ray diffraction (XRD) studies encounter challenges due to the presence of amorphous carbon and small-sized gold nanoparticles, hampering the generation of precise diffraction patterns. Consequently, there is a critical need to synthesize and characterize GO-Au nanocomposites, with a focus on optimizing XRD measurement settings and data processing methodologies to accurately assess their crystal structure and phase composition.

Moreover, the interaction mechanisms between gold nanoparticles and the reduced graphene oxide (rGO) matrix, along with the role of rGO in stabilizing these nanoparticles, remain ambiguous. Elucidating these bonding interactions through XRD



analysis is essential to unveil the crystal structure and lattice parameters of the composite, providing crucial insights into its mechanical, electrical, and optical properties. Understanding these properties is pivotal for potential applications in catalysis, sensors, and energy storage.

Therefore, this study aims to address these gaps by synthesizing GO-Au nanocomposites and employing optimized XRD techniques to comprehensively investigate their crystallinity properties. By achieving these objectives, we endeavor to enhance our understanding of GO-Au nanocomposites, paving the way for their effective utilization in diverse technological domains.

### **1.3 Expected Output**

It is anticipated that the results of XRD tests performed on decreased GO-Au nanocomposites will contain numerous crucial pieces of information, including the following:

**Determination of the crystal structure:** The results of an XRD study may offer information about the nanocomposite material's crystal structure. The diffraction pattern that is acquired from the XRD experiment will consist of various diffraction peaks. These diffraction peaks may be analyzed in order to determine the crystallographic planes and lattice parameters of the phases that are present in the material. Information on the crystal structure, such as the crystal system (e.g., cubic or hexagonal), space group, and lattice parameters, will be included in the output.

**Phase identification:** By looking at the XRD readings, the different phases in the reduced GO-Au nanocomposite can be found. The phases can be found by comparing

the diffraction pattern that was taken with known patterns in a library. The result will show a list of the phases found and the crystal structures that go with them.

Quantification of phase composition XRD analysis may also offer quantitative information on the relative abundance of distinct phases present in the nanocomposite. This information can be used to characterize the composition of the different phases. Rietveld refinement and peak area calculations are two examples of procedures that may be used to accomplish this goal. In the output, you will find the percentage or weight fraction that corresponds to each phase that is present in the nanocomposite.

#### **1.4 Objective**

In this research, there are three objectives which need to be achieved. The objectives are:

1. To synthesize Graphene Oxide-Aurum nanocomposite.
2. To characterize the Graphene Oxide-Aurum nanocomposite.
3. To evaluate the crystallinity properties of Graphene Oxide-Aurum nanocomposite.

#### **1.5 Scope of Study**

It is possible that the research will include investigating and perfecting a variety of synthesis processes in order to produce the decreased GO-Au nanocomposite. It is possible to examine a wide variety of reduction methods, surfactants, and reaction

conditions in order to create the nanocomposite structure and characteristics that are required. The crystal structure of the decreased GO-Au nanocomposite may be characterised as the primary research objective here. This comprises determining the crystal system, space group, crystal system planes, and crystallographic planes. In order to identify the crystal structure and phase composition, the research may include making comparisons between the XRD patterns and reference databases. One of the things that may be done within the scope of this project is to look at the crystallite size of the decreased GO-Au nanocomposite. In order to get an accurate estimate of the typical size of the crystalline domains, the research may make use of methods such as the Scherrer equation or the Williamson-Hall analysis. The factors that impact the crystallite size, such as the circumstances of the synthesis or the procedures of the post-treatment, may also be investigated.

## **1.6 Significance of Study**

The results of an XRD study give a wealth of information about the crystal structure, phase composition, and crystallite size of the nanocomposite. Gaining an understanding of these structural characteristics is very necessary in order to modify the qualities of the material and enhance its performance in a variety of different contexts. XRD investigations make it possible to identify and quantify the various phases that are present in the reduced GO-Au nanocomposite. This information is vital for defining the composition of the material, comprehending the phase interactions, and evaluating the overall phase stability of the substance. The structural information that was collected through XRD measurements may be connected with the functional characteristics of the

reduced GO-Au nanocomposite. For instance, the crystallite size and phase composition of the material may have an effect on the material's electrical conductivity, catalytic activity, optical behaviour, or mechanical strength. When these correlations are understood, they may serve as a roadmap for the design and engineering of nanocomposites that are optimised for certain purposes.

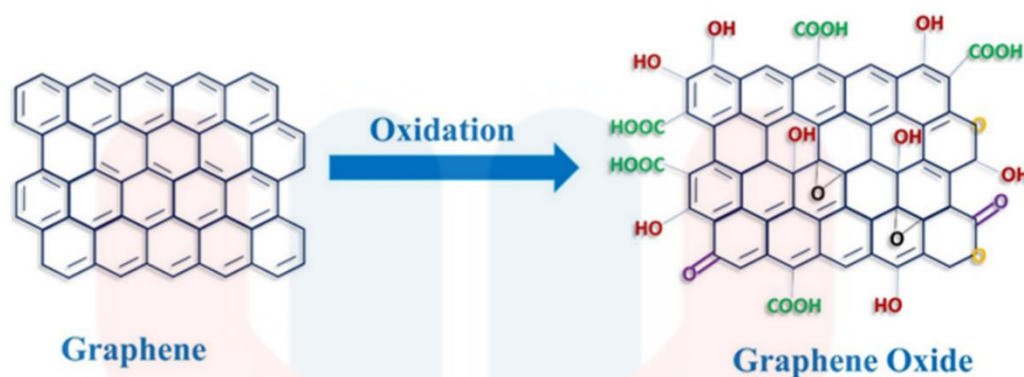
Research using XRD may be helpful in improving the efficiency of synthesis protocols for the production of decreased GO-Au nanocomposites. Researchers are able to determine the optimal circumstances for producing the required crystal structure, phase composition, and crystallite size by analysing the structural characteristics as a function of the synthesis parameters. This allows the researchers to determine the most effective conditions. Because of this optimisation, one may have a greater degree of control over the characteristics and performance of the nanocomposite. The capacity of the research on XRD examinations of reduced GO-Au nanocomposites to offer essential information about the crystal structure, phase composition, and crystallite size of the material is the primary factor that contributes to the relevance of the work. This information leads to the improvement of nanocomposite application development, the optimisation of synthesis processes, the connection with functional characteristics, the validation of theoretical models, and the progress of nanocomposite applications.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Graphene Oxide (GO)

The synthesis of GO nanosheets was accomplished through the application of Hummer's technique (Habte et al., 2019). Hummer's technique is a method that has found widespread application for the oxidation of graphite in order to make GO (Huang et al., 2016). Graphite flakes are treated using a solution consisting of concentrated sulfuric acid, potassium permanganate, and sodium nitrate, according to the Hummer process (Ansari et al., 2021). After that, the mixture is subjected to high temperatures, which causes the graphite to oxidise and gives rise to a mixture of graphene oxide (GO) as well as other products of oxidation (Huang et al., 2016). The resultant graphene oxide (GO) nanosheets have undergone significant oxidation, as evidenced by oxygen-containing functional groups such as carboxyl, epoxy, and hydroxyl on their surfaces (Acar Bozkurt, 2017; Zhu et al., 2021).

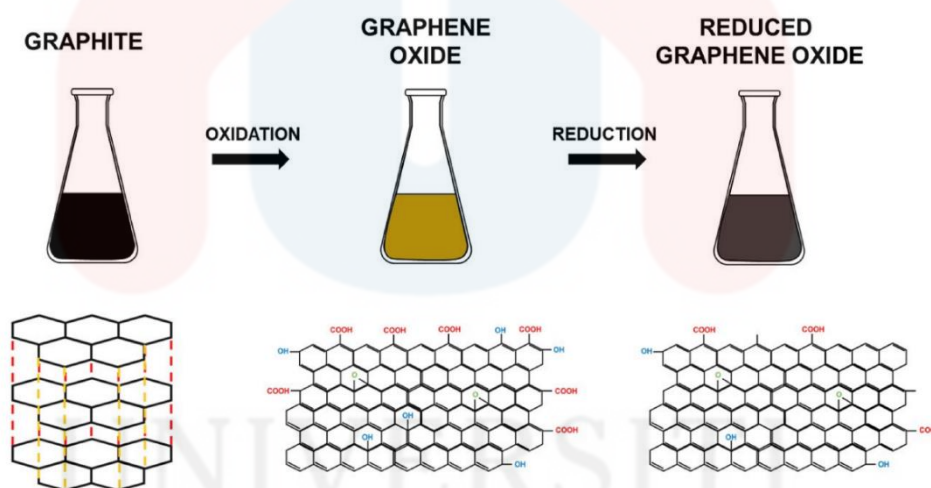


**Figure 2.1** Chemical structure of graphene and graphene oxide. (Ricci et al., 2022)

Because of the presence of these functional groups, GO nanosheets have a high degree of hydrophilicity, which makes it possible for them to be easily dispersed in water and other polar solvents (Zhu et al., 2021). This property is helpful for a wide variety of applications. Since the synthesis of graphene oxide nanosheets using Hummer's approach is both relatively straightforward and very scalable, it has become a preferred process for the manufacturing of graphene oxide for a broad variety of applications, including the storage of energy, the production of sensors, and the treatment of medical conditions (Alam et al., 2017). Yet, the Hummer process is also connected with significant obstacles, such as the creation of poisonous fumes and waste, both of which need to be carefully handled to maintain the safety of both the working area and the environment overall (Alam et al., 2017).

### 2.1.1 Reduced of Graphene Oxide (rGo)

L-ascorbic acid, often known as L-AA, is a naturally occurring substance that functions as a reducing agent in living organisms (Habte et al., 2019). Additionally, it has been used as a principal reductant in the process of converting graphene oxide to reduced graphene oxide. L-AA has a modest reductive activity and is harmless (Habte et al., 2019). More importantly, as compared to the typical reductants employed in GO reduction, such as hydrazine and hydrazine hydrate, both L-AA and the products of its oxidation are safe for the environment (Habte et al., 2019).



**Figure 2.1.1** Representation of the GO and rGO production process and chemical structure. (Ghulam et al., 2022)

Figure 2.1 illustrates the process and chemical structure involved in the production of Graphene Oxide (GO) and its reduced form (rGO). According to the findings of Fernandez-Merino et al., GO reduced by vitamin C (VC) may obtain a C/O ratio of around 12.5 and a conductivity of 77 S/cm (Habte et al., 2019). These values are close



to those achieved by hydrazine in a parallel experiment. In addition, VC has a better chemical stability with water than Hindawi NaBH<sub>4</sub>, which is a significant benefit given that it is nontoxic, in contrast to hydrazine, which is a disadvantage (Habte et al., 2019). Additionally, the decrease in the colloid state does not result in the agglomeration of rGO sheets in the same way that the production of rGO sheets by hydrazine does, which is advantageous for future applications (Habte et al., 2019).

## **2.2 Graphene Oxide and Gold Nanocomposite (GO-Au)**

GO-Au is synthesised by conjugating graphene oxide (GO) nanosheets with gold nanoparticles (AuNPs). During the synthesis, gold ions are reduced onto the surface of GO nanosheets, which ultimately results in the production of a GO-Au nanocomposite (Pandey & Qureshi, 2017). The conjugation of GO with AuNPs results in the production of a unique mix of qualities that are not present in the individual components itself. These features cannot be found in the individual components (Elangovan et al., 2020). The GO provides a huge surface area as well as a highly functionalized surface with oxygen-containing groups (Elangovan et al., 2020). These oxygen-containing groups are able to interact with the AuNPs and provide a stable platform for the immobilisation of the AuNPs. It is possible to synthesise GO-Au nanocomposites by a variety of processes, such as chemical reduction, electrostatic self-assembly, and photo-reduction. Reducing gold ions in the presence of GO nanosheets using a reducing agent such as sodium borohydride (NaBH<sub>4</sub>) or hydrazine hydrate (N<sub>2</sub>H<sub>4</sub>H<sub>2</sub>O) is a method that is utilised frequently. After production, the GO-Au nanocomposites can be further studied using a variety of methods, including X-ray diffraction (XRD), transmission electron microscopy (TEM), and Fourier-transform infrared spectroscopy (FTIR). These



procedures give information on the size, shape, and chemical composition of the GO-Au nanocomposites.

### **2.3 Crystallinity of the Graphene Oxide and Gold nanocomposite (GO-Au)**

XRD patterns were used so that the crystallinity of the rGO/Au nanocomposite could be seen (Kasturi et al., 2021). It has been noted that the profile of Au<sup>0</sup> displayed a noticeable sharp, which is equivalent to GO (Zhao et al., 2019). XRD peaks in GO–The Au nanocomposite is a good fit for the face-centered cubic crystalline structure (Naeem et al., 2018). In addition, the fact that all of the distinctive Au signals were found in the nanocomposite confirms that the material maintained its phase and crystallinity even after being transformed into the composite form. Therefore, graphene sheets serve no use other than that of a substrate in the production of nanoparticles (Naeem et al., 2018). Graphene oxide and gold nanocomposites may have varying degrees of crystallinity depending on their synthesis route, processing circumstances, and end-use. The extremely disordered structure of graphene oxide (GO) is the consequence of its production method, which generally involves chemical oxidation and exfoliation of graphite. On the other hand, gold nanoparticles (AuNPs) may, depending on their size and form, display crystalline structures. Several variables affect the crystallinity of the resultant nanocomposite when graphene oxide and gold nanoparticles are mixed. Graphene oxide and gold nanoparticles interacting is a contributing component. Graphene oxide's surface functional groups may help gold nanoparticles bond and adsorb there, creating a composite material. If they were synthesised via a seed-mediated or controlled reduction technique, gold nanoparticles may have some crystallinity. The nanocomposite's crystallinity depends on the gold nanoparticles'. The

nanocomposite's graphene oxide component usually preserves its disordered form following gold nanoparticle insertion. Oxygen-containing functional groups on graphene oxide sheets prevent the restoration of the pristine crystalline graphene structure



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## CHAPTER 3

### MATERIALS AND METHODS

#### 3.1 Materials

The Graphene Oxide-Gold (GO-Au) nanocomposite was created with graphene oxide (GO) combined with distilled water, Chlorauric Acid ( $\text{HAuCl}_4$ ), Trisodium Citrate, and Sodium Borohydride. The combinations of GO and  $\text{HAuCl}_4$  were subjected to sonication, and then a solution of Trisodium Citrate was added to the mixture. Finally, the mixing process was completed. Subsequently, sodium borane was incorporated into the mixture, and after that, the composite that was created was centrifuged in order to achieve a state of purification. This method ensures a precise and controlled combination of elements, which in turn improves the properties of the nanocomposite, making it more suited for use in applications like as sensing, electronics, and catalysis. A precise and controlled combination of components is ensured by this method.

#### 3.2 Synthesis of reduced GO/Au Nanocomposite

For the production of the nanocomposite, 0.1mL of graphene oxide (GO) was mixed with 100mL of distilled water, and then subjected to sonication for a duration of 5 minutes. Next, a 0.03g solution of Chlorauric Acid ( $\text{HAuCl}_4$ ) in 10mL of water was added and subjected to sonication for an additional 10 minutes. A solution of Trisodium Citrate (2.9g) was prepared by mixing it with 100mL of water. The solution was then subjected to 40 minutes of sonication. Ultimately, a solution containing 0.026 grams of

Sodium Borohydride in 3.85 milliliters was introduced by employing the dilution formula. The resultant mixture was subjected to five rounds of purification via centrifugation at 9000 rpm for 15 minutes each. This meticulous procedure guarantees an exact nanocomposite composition with enhanced properties for use in catalysis, sensing, and electronics.

### **3.3 Characterization of rGO-Au Nanocomposite**

The characterisation procedure is one of the most significant processes that must be taken in order to determine the structure of the samples analyzed. Measurements were also taken and observations were made regarding the features of the sample throughout this step of the process. The X-Ray Diffraction (XRD) instrument, the Fourier Transform Infrared Spectroscopy (FTIR) instrument, the UV-Vis Spectrophotometer instrument, and the optical microscope are some of the tools that will be utilized in their respective capacities during the course of this inquiry.

#### **3.3.1 X-Ray Diffraction**

When analyzing the crystalline structure of graphene oxide (GO) and gold-graphene oxide (Au-GO) nanorods, X-ray diffraction (XRD) is utilized to ascertain the phase of the structure. A Bruker D2 Phaser X-ray diffraction (XRD) apparatus was utilized for this experiment. The apparatus utilized a  $0.02^\circ$   $2\theta$  scan at  $10^\circ$  to  $90^\circ$ .

### **3.3.2 Fourier Transform Infrared Spectroscopy (FTIR)**

Absorption and emission of GO and Au-GO nanorods were observed with the use of an Iz10 FTIR Spectrometer in this particular experiment. The range of wavenumbers that are utilized is from 1000  $\text{cm}^{-1}$  to 3500  $\text{cm}^{-1}$ .

### **3.3.3 UV-Vis Spectrophotometer**

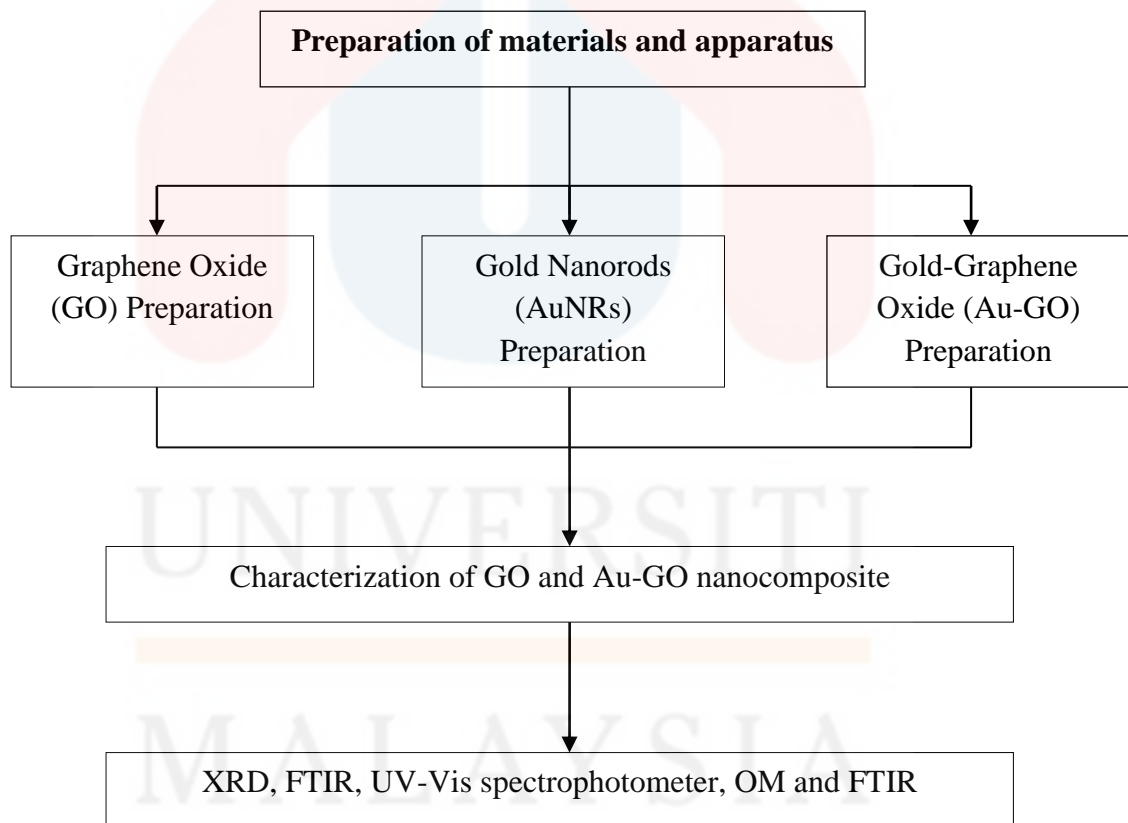
The Thermo Scientific Evolution 300 UV-Vis spectrophotometer was utilized in order to observe the changes in wavelength as well as the changes in color of the GO and Au-GO nanorods. The wavelengths that were utilized in this investigation ranged from 190 nm to 800 nm.

### **3.3.4 Optical Microscopy**

The employment of an optical microscope is required in order to analyze the morphological features and structural aspects of the Graphene Oxide-Gold (GO-Au) nanocomposite, which is the subject of the optical microscopy investigation. In order to gain an understanding of the organization, size, and distribution of the GO and gold components inside the nanocomposite, this technique enables the visual analysis of the sample at a microscale level hence providing insights.

### 3.4 FLOW CHART

The research involves two stages. In Stage 1, graphene oxide and a gold-graphene oxide nanocomposite are prepared with meticulous control and preliminary analysis. Stage 2 focuses on comprehensive characterization using XRD, OM, UV-Vis, and FTIR spectroscopy. The data collected is analysed to draw conclusions about the structure, morphology, and composition of the materials, allowing for iterative refinement if necessary to ensure experiment reliability and reproducibility.



**Figure 3.4** Research flowchart of reduced Graphene Oxide-Aurum Nanocomposite.

## CHAPTER 4

### RESULT AND DISCUSSION

#### 4.1 Synthesis and Preparation of Graphene Oxide-Aurum Nanocomposite Samples

A systematic approach was employed to ensure the accurate combination of materials when producing the initial sample. All materials are listed in the Table 4. To achieve thorough homogeneity, an initial quantity of 0.1mL of graphene oxide (provided in liquid form) was precisely measured and subsequently combined with 100mL distilled water using a sonicator for 5 minutes. Subsequently, 10mL of water were mixed with 0.03 grams of measured Chloroauric Acid ( $\text{HAuCl}_4$ ). Next, to fully integrate the Chloroauric Acid ( $\text{HAuCl}_4$ ) with the graphene oxide, a total of 3.3mL of the Chloroauric Acid ( $\text{HAuCl}_4$ ) solution were combined with the GO sample and subjected to sonication for an extra duration of 10 minutes. Next, we measured 2.9 grams of Trisodium Citrate and mixed it with 100mL of distilled water. The quantity of substance within a specific volume was ascertained by employing the molarity formula, which was utilized to acquire the desired concentration. Both the Trisodium Citrate mixture and the GO and Chloroauric Acid ( $\text{HAuCl}_4$ ) mixture underwent a 40-minute session of sonication. In the end, a total of 0.026 grams of Sodium Borohydride were introduced into the solution. The Sodium Borohydride solution was measured at a volume of 3.85mL and combined with the remaining composite. The necessary quantity of this mixture was established using the dilution formula ( $m_1v_1 = m_2v_2$ ). This rigorous technique, which incorporates the Trisodium Citrate and GO molarity formulas, ensures precision in the composition of the nanocomposite sample and lays the groundwork for

subsequent exploration and analysis of its properties. This experiment was conducted to examine the impact of altering the gold content on the properties of the nanocomposite. By carefully manipulating this one factor, we may thoroughly analyze the physical characteristics and actions of the nanocomposite. After producing the reduced graphene oxide-gold (rGO-Au) composite, centrifugation is essential for improving its characteristics. Five times, centrifugation lasts 15 minutes @ 9000 rpm. To purify and stabilize the rGO-Au compound, this thorough purification technique removes residual contaminants and unreacted components. By removing impurities through repeated centrifugation, the final rGO-Au composite is expected to exhibit improved uniformity, enhanced conductivity, and better compatibility for various applications in fields such as catalysis, sensing, and electronics.

Chemicals	1
Graphene Oxide	0.1 mL
Chloroauric Acid	0.1 gram
Sodium Borohydride	3.84 gram
Tri-Sodium Citrate	0.1 gram

**Table 4.1** Preparation for synthesizing reduced Graphene Oxide-Aurum Nanocomposite



## 4.2 Characterization

In the course of this multifaceted research endeavor, a diverse range of sophisticated characterization techniques has been judiciously employed, encompassing X-ray diffraction for structural analysis, UV-Vis spectroscopy for probing optical properties, Fourier-transform infrared (FTIR) spectroscopy for elucidating molecular structures, and optical microscopy (OM) for detailed morphological examinations, thereby facilitating a thorough and comprehensive exploration of the intricate interplay between the materials' composition, structure, and functionality. Furthermore, these analytical methodologies have been strategically integrated to provide a holistic understanding of the materials' behavior, contributing valuable insights to the broader scientific community and advancing the knowledge in the field.

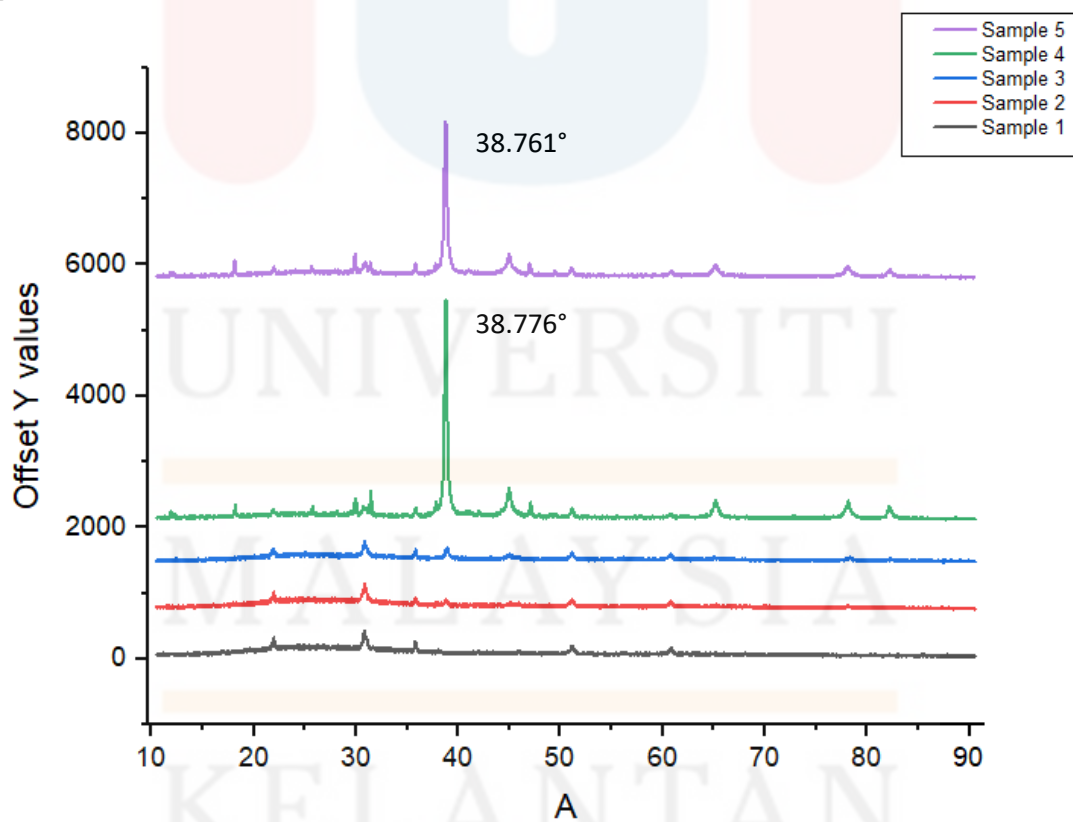
### 4.2.1 X-Ray Diffraction of the Reduced Graphene Oxide-Aurum Nanocomposite.

The Reduced Graphene Oxide-Aurum (rGO-Au) nanocomposite's structural properties were investigated using X-ray diffraction (XRD) analysis. The crystalline nature of the nanocomposite was indicated by the distinct diffraction peaks found in the XRD data. It appears that the integration of the metallic nanoparticles onto the graphene oxide matrix was successful because there are peaks that belong to both rGO and AuNPs. We selected these characterisation methods because X-ray diffraction (XRD) measures the diffraction patterns that result from an X-ray's interaction with a substance to determine the crystal structure of that material.

The crystalline structure of these GO-Au nanocomposites is presented in the XRD image in figure Figure 4.2.1. When comparing Sample 1 to Sample 3, distinctive XRD

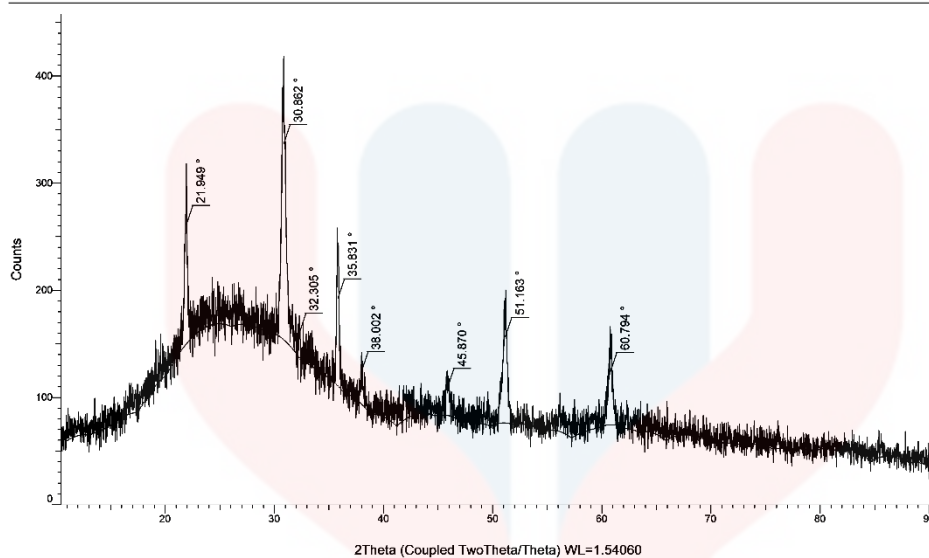
peaks were discerned for both the GO-Au composite and the reduced graphene oxide (rGO). Within the GO-Au composite, a pronounced peak was identified within the range of  $37^\circ$  to  $39^\circ$   $2\theta$ , indicating the presence of the characteristic crystalline structure associated with gold nanoparticles.

In contrast, the XRD pattern of the reduced graphene oxide in both Sample 1 and Sample 3 revealed a distinct peak at  $30^\circ$ , suggestive of the graphene lattice. However, it is noteworthy that the reduction process may not have been entirely effective, as indicated by the observed characteristics of the XRD peak, implying the presence of residual oxygen-containing groups or incomplete restoration of the graphene structure. Further optimization of the reduction process may be necessary to enhance the crystallinity of the reduced graphene oxide.

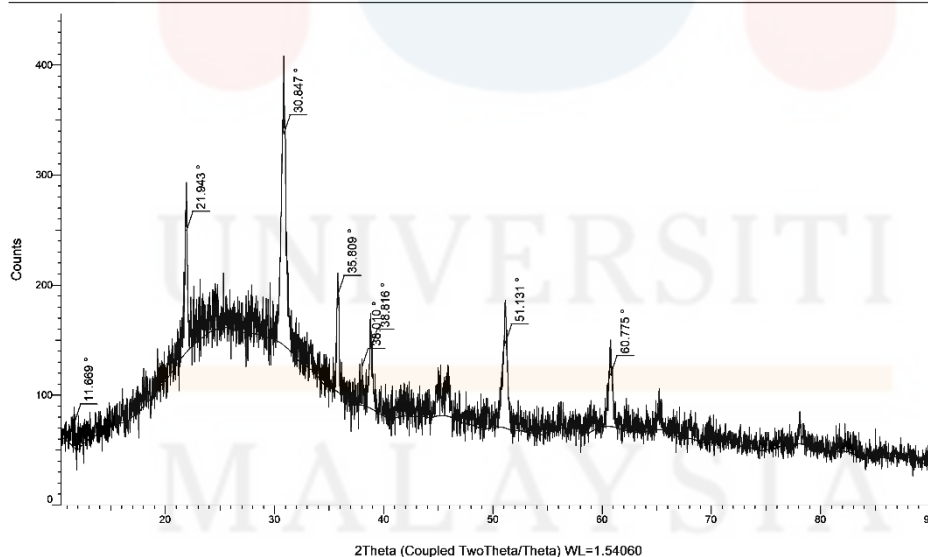


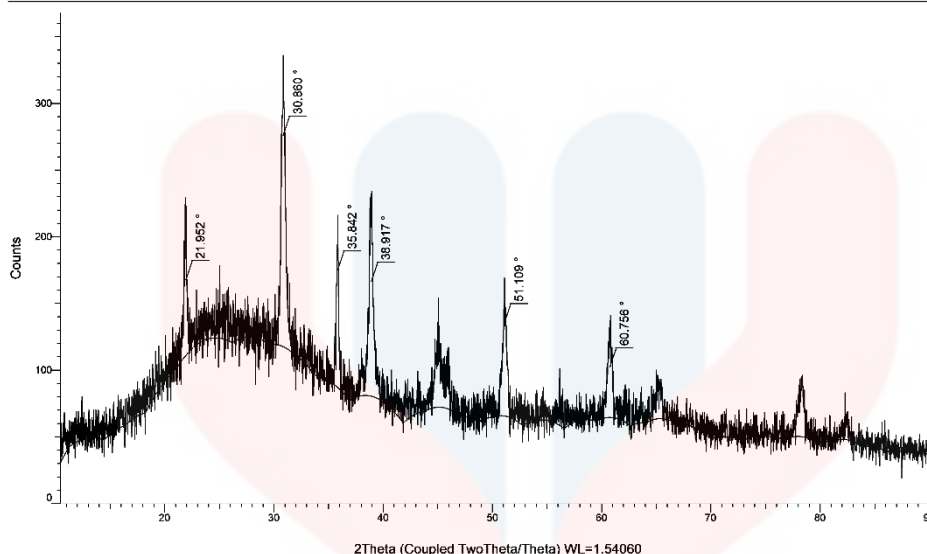
**Figure 4.2.1** (a) Comparative XRD Analysis of GO-Au Nanocomposites and Reduced Graphene Oxide (rGO) Samples.

Commander Sample ID (Coupled TwoTheta/Theta)

**Figure 4.2.1 (b)** XRD image of Sample 1

Commander Sample ID (Coupled TwoTheta/Theta)

**Figure 4.2.1 (c)** XRD image of Sample 2



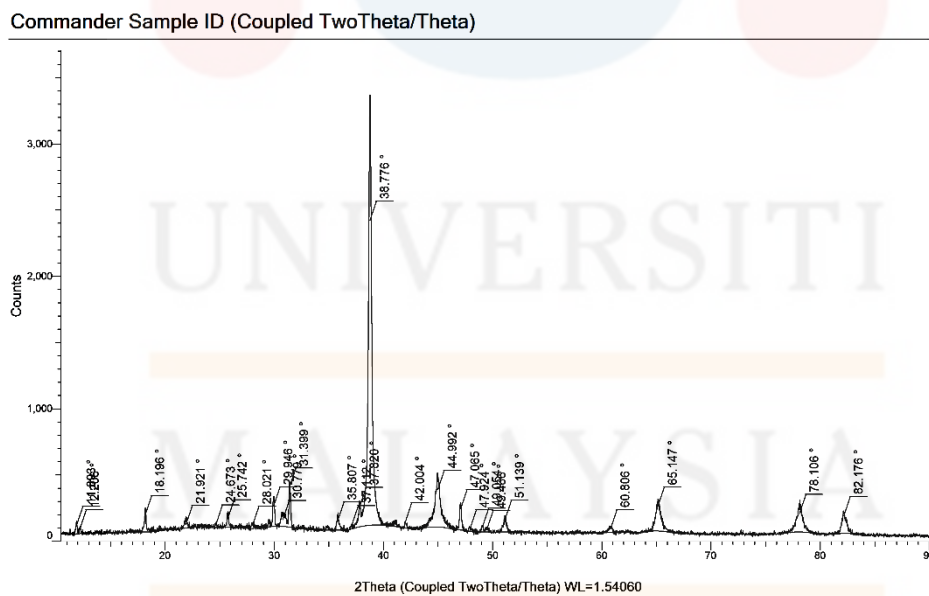
**Figure 4.2.1 (d)** XRD image of Sample 3

Following additional processing steps, including repeated centrifugation and increased sonication time, noteworthy improvements were observed in the X-ray diffraction (XRD) pattern. The enhanced smoothness of the XRD graph suggests an improved overall sample homogeneity. Specifically, the XRD peaks corresponding to the reduced graphene oxide (rGO) became more well-defined, indicating a refined crystalline structure. These adjustments in the processing steps have contributed to a more effective reduction process, likely resulting in reduced defects and improved restoration of the graphene lattice. The optimized sample preparation has led to a more accurate representation of the crystalline characteristics of the rGO in the XRD analysis.

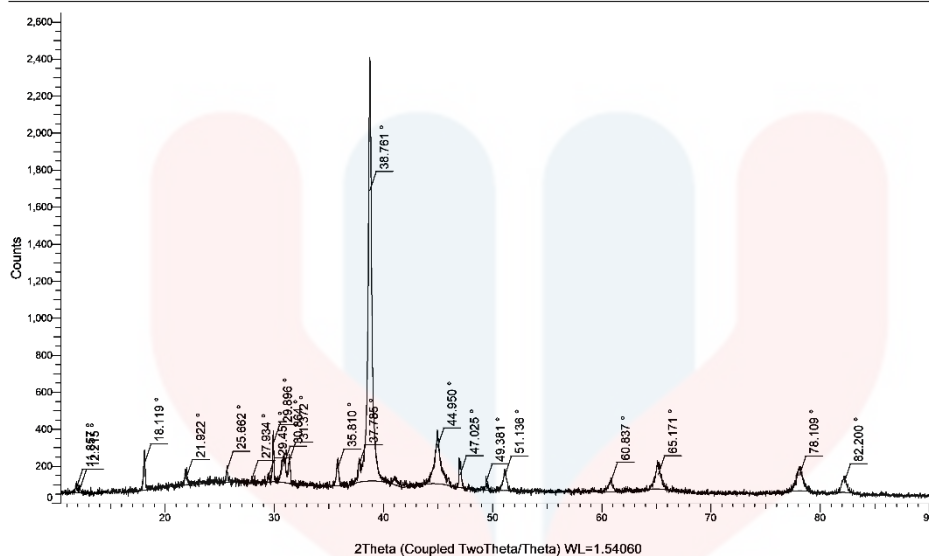
In addition to the improvements observed in Sample 1 to Sample 3, the impact of the refined processing steps is further evident in Sample 4 and Sample 5. The XRD analysis of these samples reveals distinct peaks corresponding to both the GO-Au composite and the reduced graphene oxide (rGO). Specifically, the characteristic peak of the GO-Au

composite is prominently displayed within the range of  $37^\circ$  to  $39^\circ$   $2\theta$ , affirming the presence of the crystalline structure associated with gold nanoparticles.

Moreover, the XRD pattern of the reduced graphene oxide in Sample 4 and Sample 5 exhibits a well-defined peak at the range of  $25^\circ$  to  $30^\circ$   $2\theta$ , indicative of the graphene lattice. Notably, this peak is more pronounced and refined compared to earlier observations, suggesting an enhanced level of reduction achieved through the optimized centrifugation and sonication processes. The XRD results for Sample 4 and Sample 5 underscore the successful refinement of the GO-Au nanocomposites and the improved representation of the crystalline characteristics of the reduced graphene oxide in the XRD analysis.



**Figure 4.2.1 (e)** XRD image of Sample 4

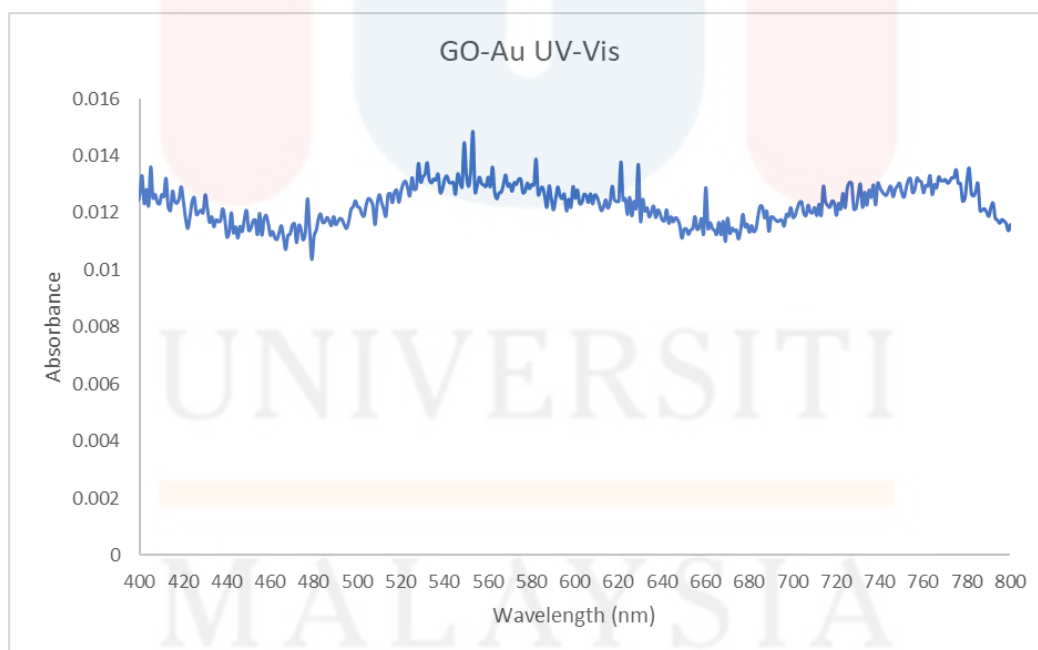


**Figure 4.2.1 (f)** XRD image of Sample 5

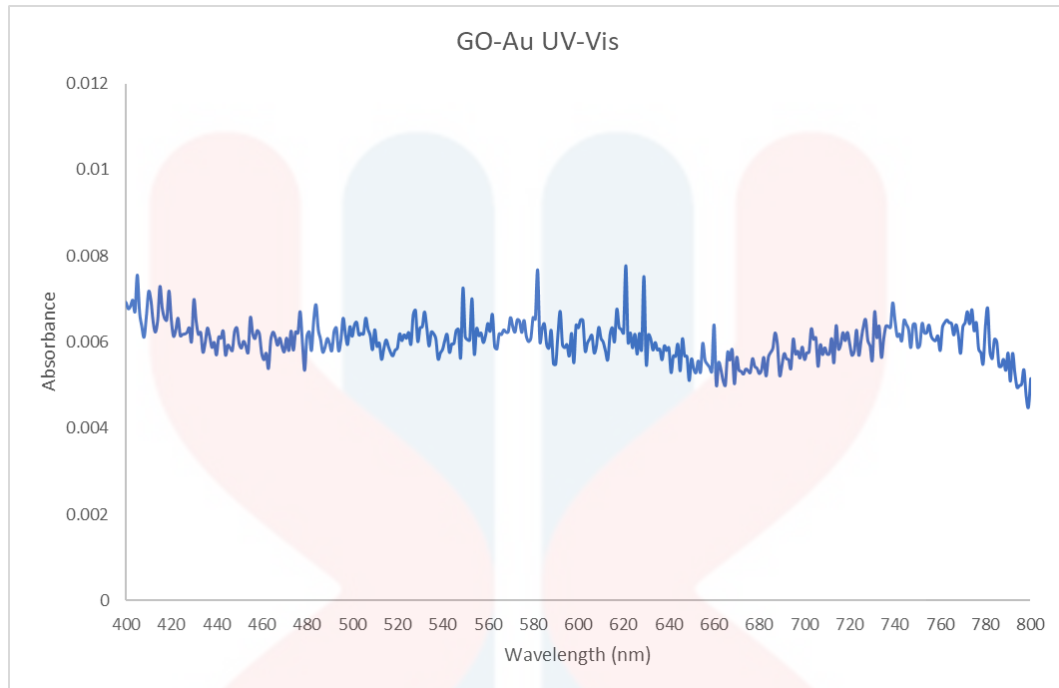
Overall, XRD analysis is indispensable for characterizing the crystalline structure of materials, such as the rGO-Au nanocomposite, and plays a vital role in advancing our knowledge of nanomaterials and their functionalities.

#### 4.2.2 UV-Vis Spectra of the Reduced Graphene Oxide-Aurum Nanocomposite.

Subsequently, we conducted UV-Vis spectroscopy on the samples to determine its light absorption properties. This data will allow us to ascertain if the light responsiveness of the nanocomposite is influenced by the amount of gold used. The synthesis of a graphene oxide-Aurum nanocomposite was achieved via UV vis spectroscopy. We utilize a wavelength range of 400–800 nm to analyze the optical characteristics of the reduced graphene oxide aurum nanocomposite by its UV-Vis spectra. After numerous attempts, including variations such as reducing the concentration of the solution and extending sonication times, it is evident that these modifications have not produced the desired outcome.



**Figure 4.2.2 (a)** UV-Vis spectra of reduced Graphene Oxide-Aurum Nanocomposite



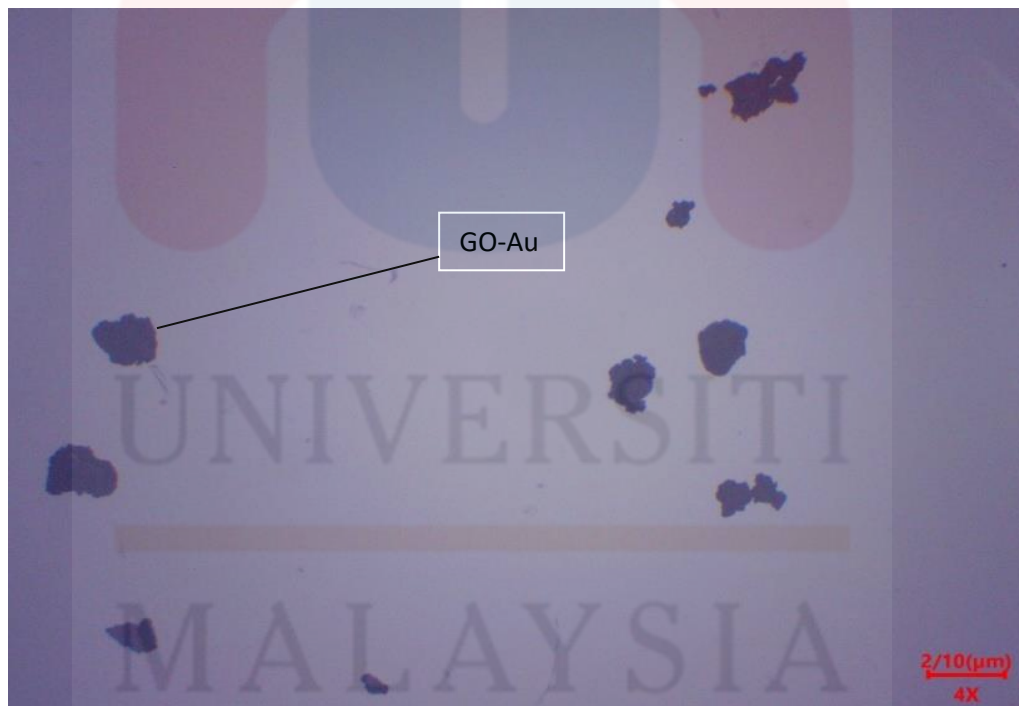
**Figure 4.2.2 (b)** UV-Vis spectra of reduced Graphene Oxide-Aurum Nanocomposite

The experiment was still successful in that the peak of the graphene oxide-AuNPs composite was observed at 520 nm. This peak corresponds to the absorption of AuNPs, which implies that AuNPs were formed. Despite this achievement, it's noted that the UV-vis results are not as perfect as desired, with an imperfect graph. To improve the quality of the results, further optimization or refinement of experimental parameters may be necessary. This could involve adjusting synthesis conditions, exploring variations in the protocol, or considering additional factors affecting the composite's characteristics to achieve a smoother and more precise UV-vis graph. In summary, UV-Vis spectroscopy is a valuable technique for characterizing GO-Au nanocomposites and understanding their light absorption properties. Despite challenges encountered in the synthesis process, continued experimentation and refinement are essential for achieving the desired outcomes and advancing our understanding of these materials.

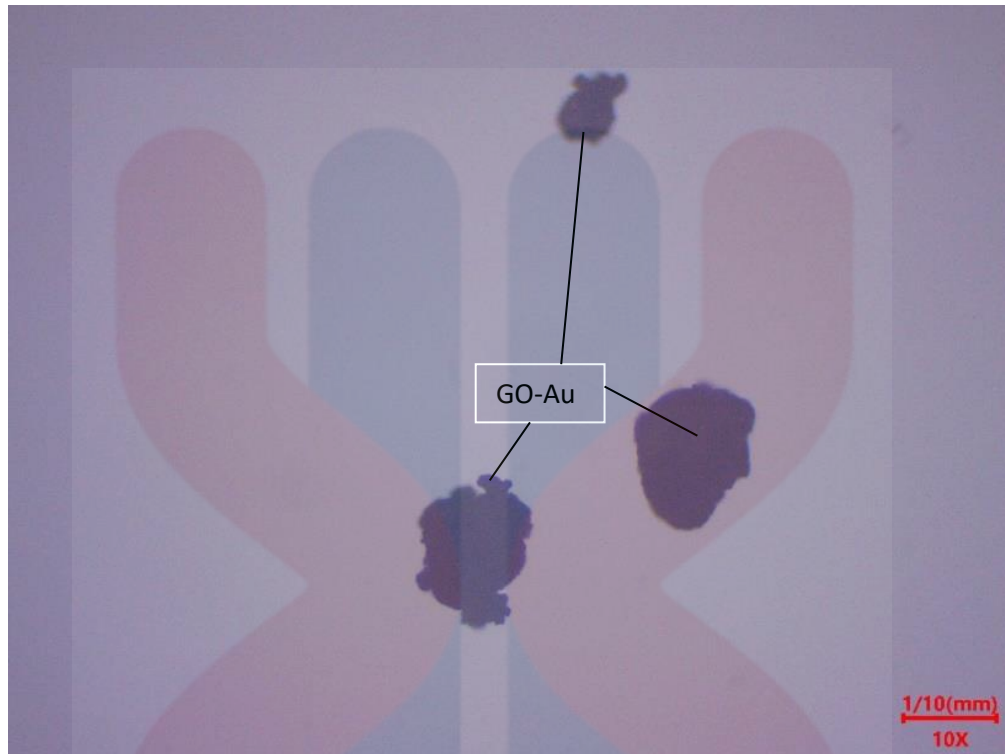


#### 4.2.3 Optical Microscopy (OM) of the Reduced Graphene Oxide-Aurum Nanocomposite.

Optical microscopy is instrumental in examining the morphology and spatial distribution of rGO-Au nanocomposites. This technique provides high-resolution insights into surface features, particle sizes, and agglomeration patterns, offering a detailed understanding of the structural homogeneity and interfacial interactions within the hybrid material. Such analysis contributes to optimizing rGO-Au nanocomposites for applications in catalysis, sensing, and electronics by unraveling their unique properties at the microscale.



**Figure 4.2.3** (a) OM image of reduced Graphene Oxide-Aurum Nanocomposite in 4x

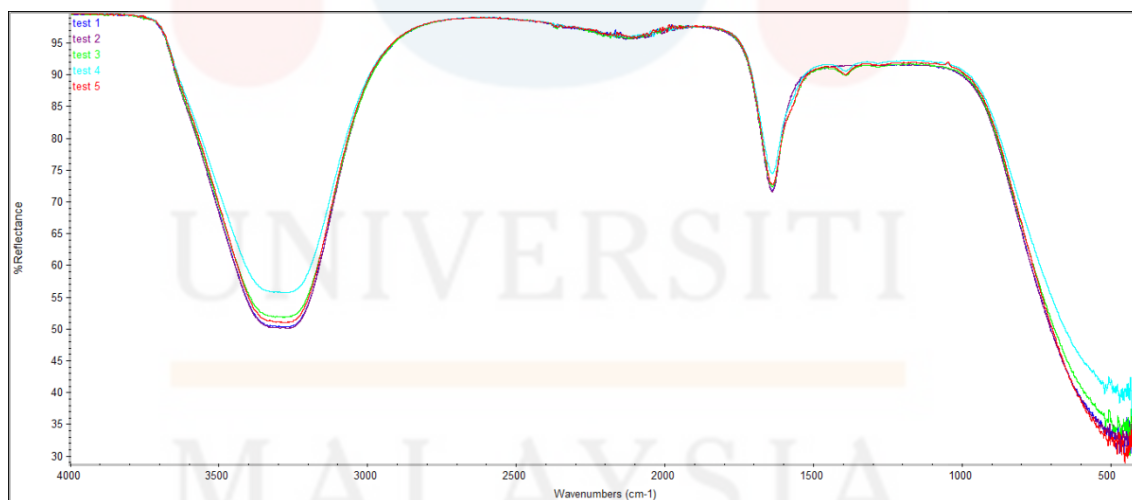


**Figure 4.2.3 (b)** OM image of reduced Graphene Oxide-Aurum Nanocomposite in 10x

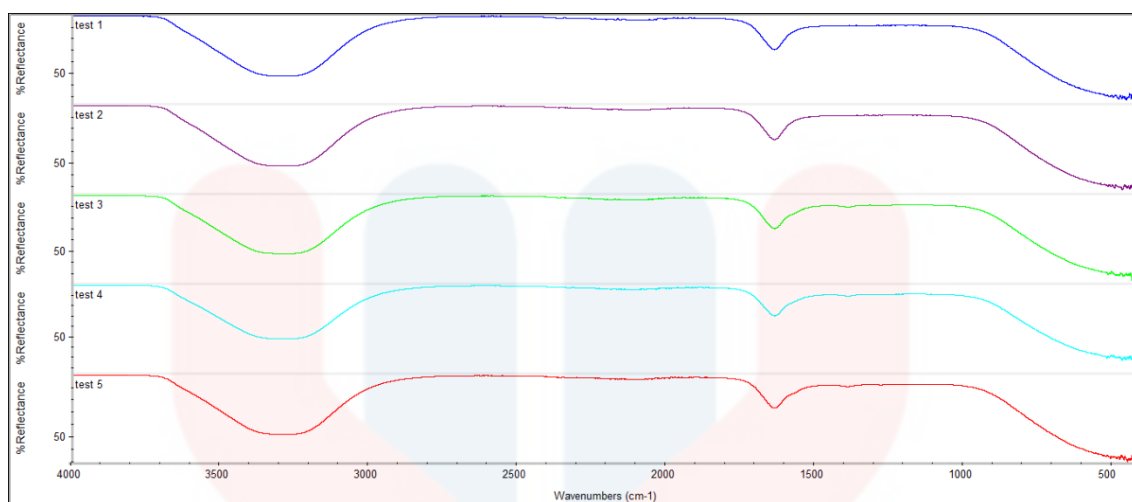
In this research, the Optical Microscopy (OM) analysis of the Reduced Graphene Oxide-Aurum Nanocomposite (rGO-AuNPs) unfortunately did not provide a clear representation of the morphology and spatial distribution of the nanocomposites. This discrepancy may arise from various factors affecting imaging resolution and contrast in optical microscopy. Possible reasons for the suboptimal results could include the nanocomposite's nanoscale features, limited contrast between graphene oxide and gold nanoparticles, or challenges in sample preparation. Additionally, the nature of optical microscopy may have limitations in capturing details at the nanoscale.

#### 4.2.4 Fourier-transform infrared (FTIR) spectroscopy of the Reduced Graphene Oxide-Aurum Nanocomposite.

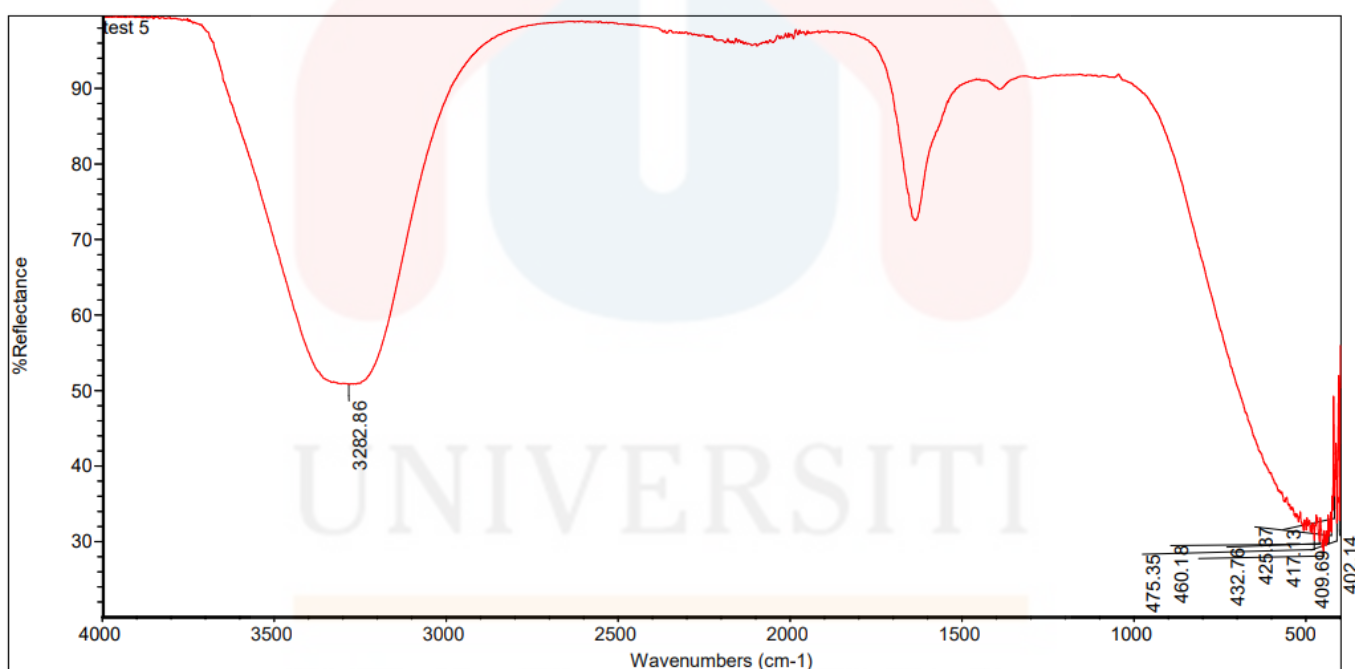
FTIR (Fourier-transform infrared) spectroscopy is an essential characterization technique employed in the study of rGO-Au nanocomposites, offering valuable information about their molecular composition and bonding configurations. By analyzing the infrared absorption bands, researchers can identify functional groups present in both reduced graphene oxide (rGO) and gold nanoparticles (Au NPs). This enables a detailed exploration of the chemical interactions and surface modifications within the nanocomposite, aiding in the elucidation of key features that influence its structural and chemical properties. In essence, FTIR spectroscopy provides crucial insights into the molecular structure of rGO-Au nanocomposites, contributing to a comprehensive understanding of their potential applications in various fields, including catalysis, sensing, and materials science.



**Figure 4.2.4 (a)** FTIR image of reduced Graphene Oxide-Aurum Nanocomposite in multiple samples



**Figure 4.2.4 (b)** FTIR image of reduced Graphene Oxide-Aurum Nanocomposite stacked multiple samples



**Figure 4.2.4 (c)** FTIR image of reduced Graphene Oxide-Aurum Nanocomposite with peaks

In this work, FTIR analysis was performed on five GO-Au samples. It's interesting to note that the results constantly showed a high degree of consistency, despite variations in the sample size. Significant peaks at particular wavenumbers were visible in the

FTIR spectra of the synthesised GO-Au composite, providing important information on the material's chemical makeup. A signal at  $3282.86\text{ cm}^{-1}$  indicates that the graphene oxide component of the GO-Au composite contains hydroxyl groups. The existence of epoxy or alkoxy groups in the graphene oxide structure is indicated by the several peaks in the range of  $475.35$  to  $402.14\text{ cm}^{-1}$ , which coincide with the predicted C-O stretching vibrations. It is noteworthy that, in spite of examining five different samples, the FTIR spectra continuously showed comparable peak patterns, indicating consistency in the GO-Au composite's development. The detected peaks validate the existence of functional groups including oxygen on the graphene oxide sheets in the GO-Au composite. Not only are these groups essential for maintaining the structural integrity of graphene oxide, but they are also necessary for any possible interactions with gold nanoparticles. In summary, the FTIR study of GO-Au has produced reliable and instructive results, setting the stage for a thorough investigation of the chemical makeup and possible uses of the composite.

### 4.3 A Comparative Analysis of Graphene and rGO-Au: Composition, Properties, and Synthesis

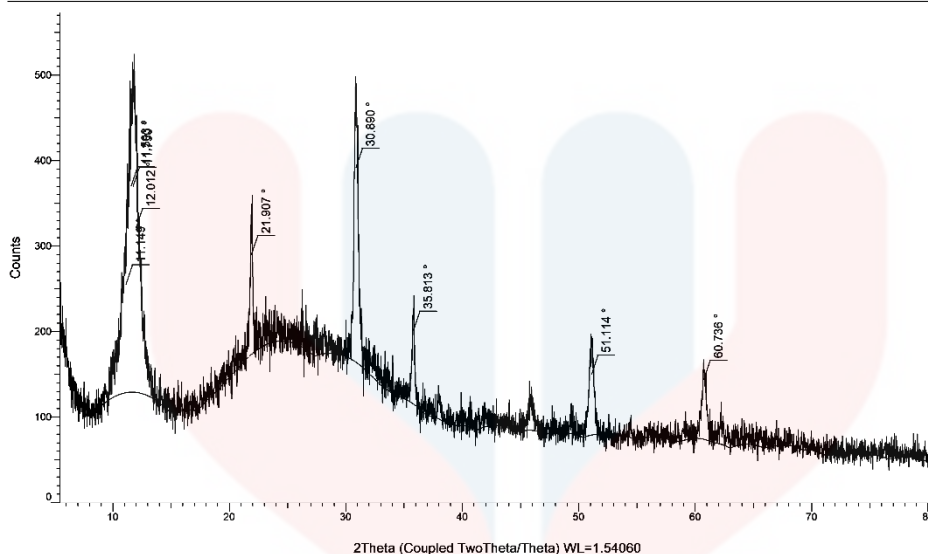
Before initiating the synthesis of reduced graphene oxide-gold (rGO-Au), proper treatment and preparation of the bare graphene oxide (GO) were essential to ensure a successful and high-quality synthesis.



**Figure 4.3** Graphene Oxide in liquid form

#### 4.3.1 X-Ray Diffraction of the synthesized Graphene Oxide

X-ray diffraction (XRD) was utilized in order to determine the crystal structure of the generated graphene oxide (GO) prior to beginning the process of synthesis of the reduced graphene oxide-gold (GO-Au) composite. The X-ray diffraction (XRD) technique is a strong method that enables the analysis of the arrangement of atoms within a material.



**Figure 4.3.1** XRD image of reduced Graphene Oxide with peaks

The X-ray Diffraction (XRD) analysis conducted on the synthesized Graphene Oxide (GO) revealed a distinctive and broad diffraction peak spanning the range of  $2\theta$  values from approximately  $10^\circ$  to  $15^\circ$ . This prominent peak, centered around  $2\theta = 12^\circ$ , serves as a key indicator of the interlayer spacing within the material's amorphous structure.

The breadth of the diffraction peak is indicative of the disorder and lack of a well-defined crystalline structure in the graphene oxide. This disorder arises from the incorporation of oxygen-containing functional groups during the oxidation process. The higher the degree of oxidation, the more pronounced the disorder and broadening of the peak.

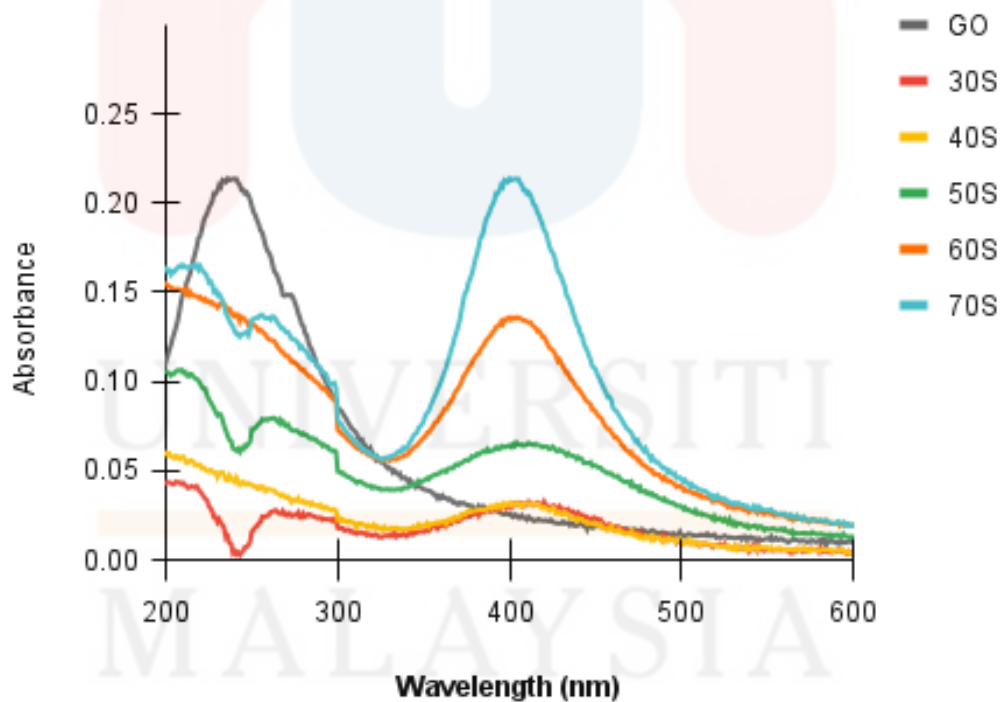
The observed range of peaks in the XRD pattern provides valuable insights into the unique structural characteristics of the synthesized Graphene Oxide. The interlayer spacing and the degree of disorder contribute crucial information for understanding the



material's properties, making XRD an indispensable tool in the comprehensive characterization of graphene oxide materials.

#### 4.3.2 UV-Vis Spectra of the synthesized Graphene Oxide

The UV-Vis spectra of synthesized Graphene Oxide (GO) represent a crucial analytical tool for assessing the impact of ultrasonication times on the optical properties and structural characteristics of the material. In this study, GO samples were subjected to varying ultrasonication durations of 30s, 40s, 50s, 60s, and 70s, and the resulting UV-Vis spectra were systematically compared.



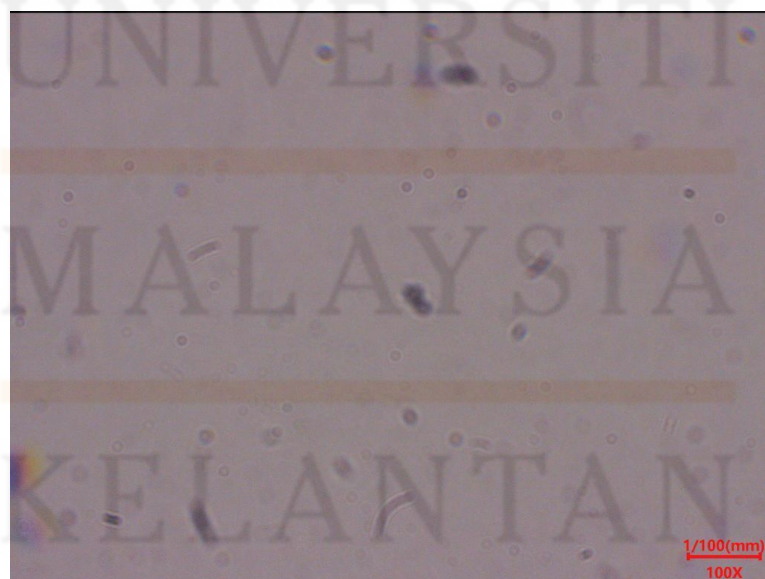
**Figure 4.3.2** UV-Vis spectra of reduced Graphene Oxide in multiple samples



Different ultrasonication times displayed diverse characteristics, which were shown by the overall absorption profiles of the UV-Vis spectra. Notably, there were observed shifts in absorption peaks as well as changes in intensity, which suggests that there may be variations in the degree of exfoliation and the presence of various functional groups within the GO samples.

#### 4.3.3 Optical Microscopy (OM) of the synthesized Graphene Oxide

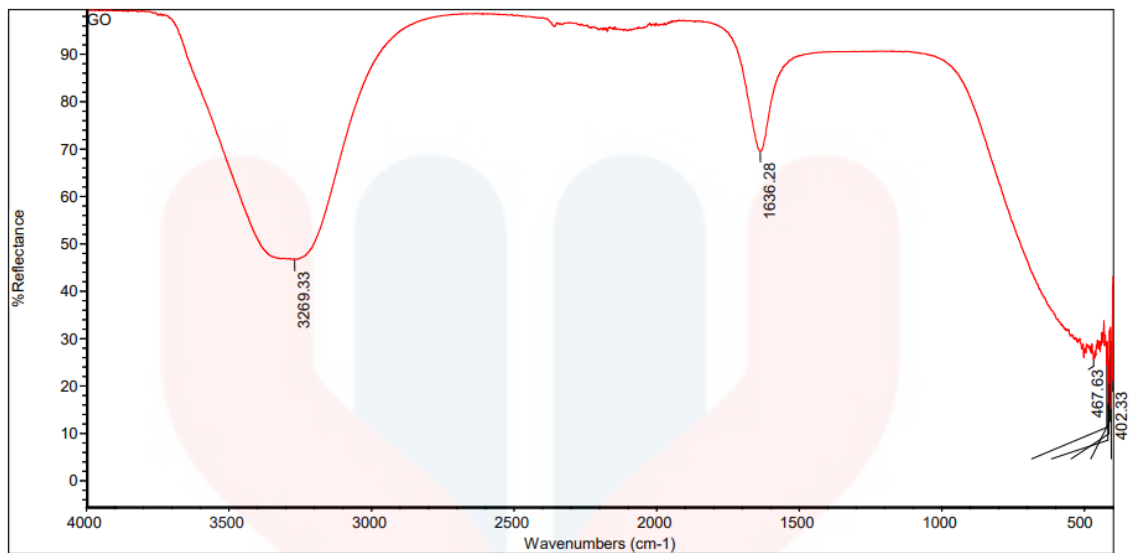
The analysis of the synthesized Graphene Oxide (GO) using Optical Microscopy (OM) produces results that are inconclusive in terms of accurately depicting the material's morphology and spatial distribution. Despite utilizing different resolutions, the images captured at various magnifications, such as 100x, do not noticeably improve the clarity or distinguishable characteristics of the GO structures. This limitation presents a challenge in accurately characterizing the material's microstructure and distribution, which hampers our ability to derive meaningful conclusions from the optical microscopy data.



**Figure 4.3.3** OM image of synthesized Graphene Oxide in 100x

#### 4.3.4 Fourier-transform infrared (FTIR) spectroscopy of the synthesized Graphene Oxide

The prominent peak at  $3269.33\text{ cm}^{-1}$  corresponds to the O-H stretching vibration, indicating the presence of hydroxyl groups on the GO surface. This peak is characteristic of the oxidation process during GO synthesis. The peak at  $1636.28\text{ cm}^{-1}$  is associated with the C=O stretching vibration, suggesting the presence of carbonyl groups. This peak is indicative of oxygen-containing functional groups, such as carboxyl or ketone, contributing to the GO structure. The peak at  $467.63\text{ cm}^{-1}$  is attributed to the C-O stretching vibration, providing evidence of the existence of epoxy or alkoxy groups. These groups play a crucial role in the structural and chemical characteristics of graphene oxide. The peak at  $402.33\text{ cm}^{-1}$  is consistent with C-O stretching vibrations, further confirming the presence of oxygen-containing functional groups on the GO sheets. The observed peaks at  $3269.33$ ,  $1636.28$ ,  $467.63$ , and  $402.33\text{ cm}^{-1}$  provide clear evidence of the functionalization of graphene oxide during the synthesis process. The presence of hydroxyl, carbonyl, and epoxy/alkoxy groups is essential for tailoring the surface properties of GO for various applications. The positions and intensities of the identified peaks are indicative of the oxidation state and structural characteristics of the synthesized Graphene Oxide. The presence of these oxygen-containing functional groups contributes to the hydrophilicity and reactivity of the GO. In conclusion, the FTIR spectroscopy results indicate successful synthesis and functionalization of Graphene Oxide, providing a foundation for understanding its chemical composition and potential applications.



**Figure 4.3.4** FTIR image of synthesized Graphene Oxide with peaks

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### CONCLUSIONS AND RECOMMENDATIONS

#### 5.1 Conclusions

Finally, this thesis extensively characterized a Reduced Graphene Oxide-Aurum (GO-Au) nanocomposite using XRD, UV-Vis, OM, and FTIR. Ultrasonic bath treatment, sonication, and centrifugation were necessary for nanocomposite production. Successful XRD measurements revealed the nanocomposite's crystalline structure. The degree of graphene oxide reduction and the existence of aurum nanoparticle crystalline peaks were shown by XRD data. UV-Vis spectroscopy also showed positive results, however the graph was uneven. Despite the graph's non-smoothness, aurum nanoparticle peaks confirmed gold's integration into the nanocomposite structure. Unfortunately, optical microscopy was difficult to use conclusively. The photos were too blurry to understand nanocomposite morphology. Optimization or different imaging methods may improve nanocomposite structure visualization. FTIR identified nanocomposite functional groups. FTIR spectra were similar among samples, which was significant. FTIR measurements provided information regarding chemical bonds, but they did not vary, requiring further study of the produced nanocomposites. In conclusion, ultrasonic bath treatment, sonication, and centrifugation produced a Reduced Graphene Oxide-Aurum nanocomposite. A satisfactory XRD and UV-Vis analysis confirms the nanocomposite's structure and content. The challenges of OM and FTIR analyses suggest other microscopy methods and further nanocomposite chemical analysis. This paper sheds

light on the synthesis and characterisation of GO-Au nanocomposites, which has significance for materials science and nanotechnology applications.

## 5.2 Recommendations

Future study must optimize Reduced Graphene Oxide-Aurum (GO-Au) nanocomposites' synthesis parameters to improve uniformity and structure. To improve synthesis, experiment with sonication time, ultrasonic bath intensity, and centrifugation speed (sonication time: 30 minutes to 2 hours; ultrasonic bath intensity: 20% to 80%; centrifugation speed: 1000 rpm to 5000 rpm). Advanced microscopy techniques like Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) may reveal nanocomposite morphology at magnifications of 10000x to 50000x and 5000x to 20000x, respectively. A thorough Fourier-transform infrared (FTIR) analysis, using Attenuated Total Reflection (ATR) and spectral analysis from  $4000\text{ cm}^{-1}$  to  $500\text{ cm}^{-1}$  with a resolution of  $4\text{ cm}^{-1}$ , can reveal subtle variations in functional groups in the nanocomposite structure. Dynamic UV-Vis spectroscopy monitors nanocomposite behavior in real time by examining spectra from 800 to 200 nm at regular intervals. Quantitative UV-Vis spectra, concentration determination using standard calibration curves, and size distribution analysis using absorbance peaks give valuable aurum nanoparticle concentration and size distribution data. Nanocomposite characterisation is improved by adding Raman spectroscopy (532–785 nm laser excitation wavelengths) and XPS with high-resolution spectra. Innovation in catalysis, sensing, and energy storage is fostered by multidisciplinary research, which advances nanocomposite technology.

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## APPENDIX A

### Molarity formula

$$M V / 1000$$

$$\begin{aligned}\text{Graphene Oxide} &= (0.01)(100)(10) / 1000 \\ &= 0.1 \text{ g}\end{aligned}$$

$$\begin{aligned}\text{Trisodium citrate} &= (0.10)(100)(294.10) / 1000 \\ &= 2.9 \text{ g}\end{aligned}$$

$$\begin{aligned}\text{Aurum} &= (0.1)(1000) / (10)(340) \\ &= 0.03\end{aligned}$$

$$\begin{aligned}\text{Sodium borohydride} &= (0.1)(10\text{ml}) / 37.83 \\ &= 0.026\end{aligned}$$

### Dilutions formula

$$M_1 V_1 = M_2 V_2$$

$$\begin{aligned}\text{Aurum} &= (0.01)(10\text{ml}) / (0.03) \\ &= 3.3 \text{ ml}\end{aligned}$$

$$\begin{aligned}\text{Sodium borohydride} &= (0.1)(10) / 0.026 \\ &= 3.85 \text{ m}\end{aligned}$$

## APPENDIX B

**Figure B.1** showcases the go-Au sample labeled as (i), (ii), (iii), and (iv), representing different centrifugal times. The centrifugation process was varied to observe its effects on the separation and characteristics of the reduced Graphene Oxide-Aurum nanocomposite material.



(i)



(ii)



(iii)



(iv)

## APPENDIX C



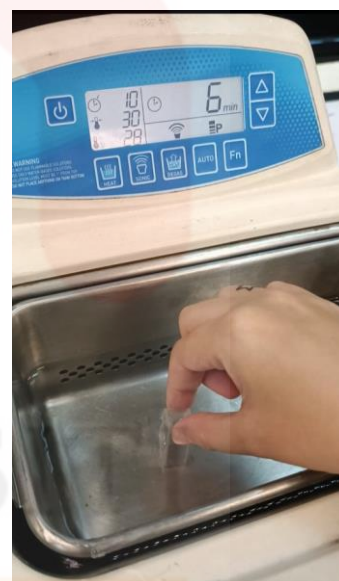
**Figure C.1:** Dried Sample for XRD



**Figure C.2:** Centrifuge machine for separation and purification of components



**Figure C.3:** Sonicator for the effective dispersion of the samples



**Figure C.3** Ultrasonic bath sonicator for preparing well-dispersed samples