

SYNTHESIS AND CHARACTERISATION OF rGO-COPPER OXIDE NANOCOMPOSITE

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Science in Chemistry

By

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of SRI DHARSHNI.J (39030037), DHARANI.R (39030006), and SOWMIYA.S (39030036), who carried out the project, entitled “**SYNTHESIS AND CHARACTERIZATION OF rGO-COPPER OXIDE NANOCOMPOSITE**” Under our supervision from 01/11/2021 to 18/04/2022.

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I, **SRI DHARSHNI. J, DHARANI. R, SOWMIYA. S**, hereby declare that the Project Report entitled “**SYNTHESIS AND CHARACTERIZATION OF rGO-COPPER OXIDE NANOCOMPOSITE**” done by me under the guidance of **Dr. S. SUNITHA, M.SC., M.PHIL., PH.D.PROFESSOR**, and **Dr. G. MURUGADOSS, M.SC., PH.D., D E P A R T M E N T O F CHEMISTRY, SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY, CHENNAI – 600119, India** is submitted in partial fulfillment of the requirements for the award of **Bachelor of Science in Chemistry** in April 2020.

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SYNTHESIS AND CHARACTERIZATION OF rGO-COPPER OXIDE NANOCOMPOSITE

VISION

*“TO EXPLORE NEW FRONTIERS IN CHEMICAL SCIENCE THROUGH
INNOVATION AND COLLABORATION”*

ABSTRACT

In this present work, Reduced Graphene Oxide (rGO) was synthesized by “MODIFIED HUMMER’S METHOD”. We developed a synthesis route for multifunctional (rGO-CuO) Nanocomposite. The rGO Nanoparticles were decorated on the surface of CuO. The Formation, Chemical and structural features of rGO-CuO nanocomposites are probed by (Fourier Transform Infrared Spectroscopy) FTIR to study the chemical bonds in the molecule by producing an infrared absorption spectrum, (X-Ray Diffraction) XRD to study the Crystallographical structure, chemical composition and physical properties of materials, (Scanning Electron Microscope) SEM to study the Morphology of the given sample and (Ultraviolet Spectroscopy) UV-Vis analysis to study the bandgap and absorption of the given sample.

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SYNTHESIS AND CHARACTERISATION OF rGO-COPPER OXIDE NANOCOMPOSITE

CHAPTER – 1

INTRODUCTION

Graphene sheets comprise a 2D layer of sp_2 -hybridized carbon atoms, arranged in a hexagonal lattice. In graphite, adjacent graphene layers are arranged with overlapping p_z orbitals. Synthesis of graphene oxide (GO) is achieved by placing graphite in concentrated acid in the presence of an oxidizing agent [1-2]. Consequently, new approaches for effectively converting GO to reduced graphene oxide (rGO) under mild conditions needed to be explored [3]. This aim of this study is to synthesize reduced graphene oxide by the Hummer's method using the cheapest material, that is, graphite, as a starting material using different parameters such as the effect of concentration, temperature, and reaction time in the oxidation level of graphene oxide in a cost effective way. This was mainly carried out using sulfuric acid, and potassium permanganate excluding sodium nitrate that emits toxic gases into the environment. This makes the method environmental friendly. Another most cost-effective and environmental friendly method is the reduction of graphene oxide into rGO (reduced graphene oxide). As well as, it was not performed with different parameters. In this project, graphene oxide has been produced with different parameters

1.1 NANOPARTICLE

A nanoparticle or ultrafine particle is usually defined as a particle of matter that is between 1 and 100 nanometer (nm) in diameter. The term is sometimes used for larger particles, up to 500 nm, or fibers and tubes that are less than 100 nm in only two directions [4]. At the lowest range, metal particles smaller than 1 nm are usually called atom clusters instead. Being much smaller than the wavelengths of visible light (400-700 nm), nanoparticles cannot be seen with ordinary optical Microscopes, requiring the use of electron microscopes or microscopes with laser. For the same reason, dispersions of nanoparticles in transparent media can be transparent [5].

The properties of nanoparticles often differ markedly from those of larger particles of the same substance. Since the typical diameter of an atom is between 0.15 and 0.6 nm, a large fraction of the nanoparticle's material lies within a few atomic diameters of its surface. Therefore, the properties of that surface layer may dominate over those of the bulk material. This effect is particularly strong for nanoparticles dispersed in a medium of different composition since the interactions between the two materials at their interface also becomes significant [6]. Nanoparticles have other physical properties that must be measured for a complete description, such as size, shape, surface properties, crystallinity, and dispersion state. Additionally, sampling and laboratory procedures can perturb their dispersion state or bias the distribution of other properties [7- 8].

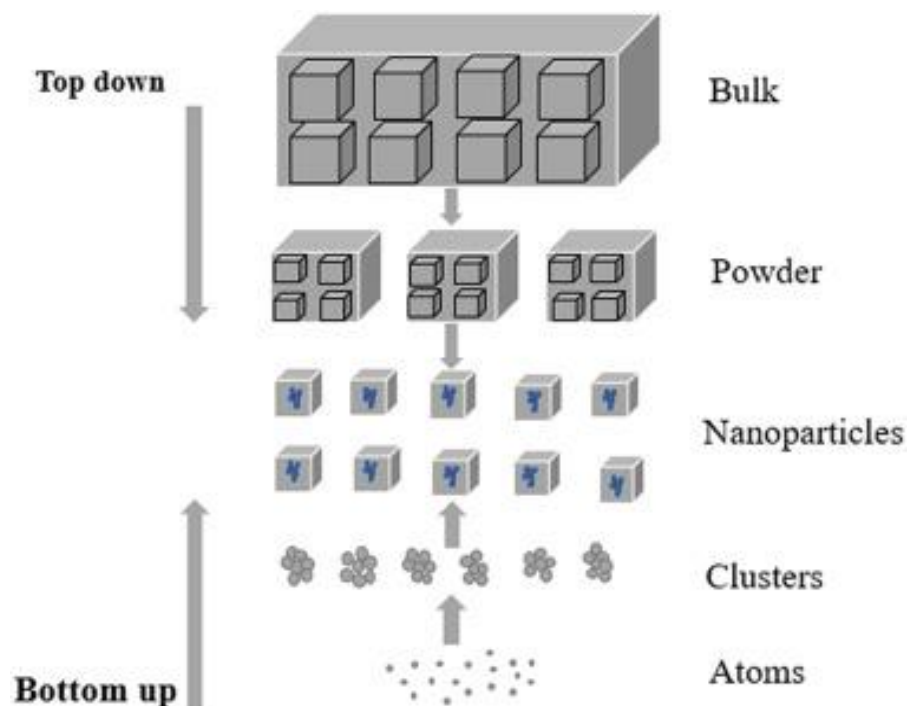


Figure 1.1 Synthesis of Nanoparticle

Nanoparticles are typically synthesized from a top-down or bottom-up approach. A bottom-up approach relies on nucleating atomic-sized materials into the eventual nanoparticles. While the exact synthesis method depends on the material being generated, some common methods include the gas phase synthesis, block copolymer synthesis, and more recently, microbial synthesis [9-10-11].

Bottom-up synthesis methods are often termed “wet” methods since they involve batches of solvents and other chemicals. Additionally, the particles often must be stabilized or capped in solution to ensure that they do not continue to grow past the nanoscale range. The particles then usually need to be moved or transferred from their solutions for their application or characterization. This can be accomplished by drop-casting the solution onto the substrate of interest.

However, in some applications, such as catalysis, it may also be necessary to remove the stabilizers from the surface of the nanoparticles after they have been immobilized on the final support of interest. The removal process can prove difficult or even impossible, leading these particles to be unusable for their intended purpose.

TOP-DOWN APPROACH

In the physical methods, mechanical methods offer the least expensive ways to produce nanomaterials in bulk (break the particles into nanostructures). But chemical fabrication methods are always easy to upscale and many, such as anodizing, are widespread industrial processes [12]. Top-down approach is the process of making nanostructures that start with larger structures and break away to nanosize to form nanomaterials. To obtain nanoscale structures in this method, first, a large object that is (2–3) orders larger in one or two dimensions than the nanoscale desired is fabricated and then nanopatterning techniques are utilized to achieve smaller features. Top-down methods actually was developed firstly by and has been widely used in microelectronics industry. Methods of deposition and nanopatterning of thin films are more advanced, and this approach has been pushed further into the regime of nanofabrication [13]. Also, applying the top-down assembly process of nanocomponents over large areas is difficult and expensive.

BOTTOM-UP APPROACH

The building of nanostructures starting with small components such as atoms or molecules is called bottom-up approach. The bottom-up techniques make use of self-processes for ordering of supramolecular or solid-state architectures from the atomic to the mesoscopic scale. The methods of bottom-up include gas-phase and liquid-phase methods. For two methods, fabrication of nanomaterials was controlled when starting from the single atom or molecule. Chemical vapor deposition (CVD) and plasma arcing are called gas-phase methods, whereas liquid-phase (LP) represented by the most established method is sol-gel synthesis. Also, a new method called molecular self-assembly emerged.

The areas of application for nanotechnology have different fields such as photonics, electronics, chemical sensors, biological sensors, and energy storage, and catalysis nanomaterial requires the manipulation into functional materials and devices. Self-assembly is the method important for designing and controlling the bottom-up assembly of the materials in the nanoscale range into structures of sheets, tubes, wires, nanoelectronic devices and drug delivery systems [14].

1.1.1 TYPES OF NANOPARTICLES

Generally Nanoparticle are classified into different types according to the size, morphology, physical and chemical properties. Some of them are carbon-based nanoparticles, ceramic nanoparticles, metal nanoparticles, semiconductor nanoparticles, polymeric nanoparticles and lipid-based nanoparticles.

CARBON-BASED NANOPARTICLES

Carbon-Based nanoparticles includes two main materials: carbon nanotubes (CNTs) and fullerenes. CNTs are nothing but graphene sheet rolled into a tube. These material are mainly used for the structural reinforcement as they are 100 times stronger than steel. Carbon nanoparticles can be used for the delivery of drugs at a cellular level. Carbon based nanoparticles are produced and used in many industrial sector. These materials include carbon nanotubes, Carbon nanofibers, carbon black, and Carbon-onions.

It has extraordinary electrical conductivity, heat conductivity and mechanical properties. They are composed of pure carbon, therefore, exhibiting high stability, good conductivity, low toxicity, environmental friendliness.

CERAMIC NANOPARTICLES

Ceramic nanoparticles are primarily made up of oxides, carbides, phosphates and carbonates of metals and metalloids. It has been successfully used as a drug delivery system against a number of diseases, such as bacterial infections, glaucoma, etc., and most widely, against cancer.

They were formed using a process called sol-gel which mixes nanoparticles with in a solution and gel to form the nanoparticles. Later methods involved sintering (Pressure and Heat). The material is so small that it has basically no flaws. Larger scale materials have flaws that render them brittle. This has some various terms of properties in electrical and magnetic physics phenomenon like Dielectric, Ferroelectric, Piezoelectric, Pyroelectric, Ferromagnetic, Magnetoresistive, Superconductive, Electro-optical.

METAL NANOPARTICLES

Metal nanoparticles are in general, one of the most common nanotechnology-based

Approaches for enveloping biosensors, due to their simplicity, physiochemical malleability and high surface areas. They can measure between 1 to 100 nm in diameter have different shape and can be composed of one or more inorganic compounds, metal nanoparticles have been used for several biomedical applications such as anticancer, radiotherapy enhancement, drug delivery, thermal ablation, antibacterial, diagnostic assays, antifungal, gene delivery, and many others.

SEMI-CONDUCTOR NANOPARTICLES

Semiconductor Nano crystals (NCs) are made from a variety of different compounds. They are referred to as II-VI, III-V or IV-VI semiconductor Nano crystals, based on the periodic table groups into which these elements are formed.

For example, silicon and germanium are group IV, GaN, GaP, GaAs, InP and InAs are III-V, while those of ZnO, ZnS, CdS, CdSe and CdTe are II-VI semiconductors. The development of “Nano chemistry” is reflected in an immense number of publications on the synthesis of semiconductor nanoparticles [15].

For instance, the spatial quantum confinement effect results in significant change in optical properties of semiconductor nanomaterials. The very high dispersity (high surface-to-volume ratio), with both physical and chemical properties of the semiconductor has a major influence on their optical and surface properties. As a result, semiconductor nanomaterials have been the focus of research for about 20 years and have attracted significant interest in research and applications in diverse disciplines such as solid-state physics, inorganic chemistry, physical chemistry, colloid chemistry, materials science, and recently biological sciences, medical sciences, engineering, and interdisciplinary fields

POLYMERIC NANOPARTICLE

The various biodegradable polymers commonly used in the fabrication of polymeric nanoparticles include poly (lactide) (PLA), poly (lactide-co-glycolide) (PLGA) copolymers, poly (ϵ -caprolactone) (PCL), and poly (amino acids) and also some natural polymers like alginate, chitosan, gelatin, and albumin. Polymer nanoparticles have favorable characteristics in terms of simple elaboration and design, good biocompatibility, broad-structure variety, and notable bio imitative characteristics.

This is expressly shown in smart drug delivery where polymer nanoparticles have a marked role since they are able to bring therapeutics right into the intended position in the human body with excellent efficiency. Most important advantages offered by the polymer nanoparticles include the following: (1) provide controlled release to the desired site, (2) provide stability to labile molecules (e.g., proteins), and (3) provide ability to modify surfaces with ligands for stealth and targeted drug delivery purposes.

LIQUID-BASED NANOPARTICLES

Liquid-Based nanoparticles (LBNPs) such as liposomes, solid liquid nanoparticles (SLN) and nanostructured liquid carriers (NLC) have received great attention in drug discovery and cancer treatment. These nanoparticles can transport hydrophobic and hydrophilic molecules, display very low or no toxicity, and increase the time of drug action by means of a prolonged half-life and a controlled release of the drug [16].

The liquid nucleic acid nanoparticles were prepared from a liquid Nano phase containing water and a water miscible organic solvent where both liquid and DNA are separately dissolved by removing the organic solvent, stable and homogeneously sized liquid-nucleic acid nanoparticle (70-100 nm) were formed [17].

1.1.2 APPLICATION OF NANOPARTICLES

Considering the unique properties discussed in section 5, Nanoparticles can be used in variety of applications. Some important of these are given below:

- I. Application in drugs and medication
- II. Application in manufacturing and materials
- III. Application in the environment
- IV. Application in electronics
- V. Application in energy harvesting
- VI. Application in mechanical industries

1.2 GRAPHENE

Graphene can be considered to be a mono molecular layer of graphite. The carbon atoms in graphene are sp^2 bonded and form a hexagonal two dimensional lattice and possess bond strength of 0.142nm. Initially, the single-layer graphene sheet was isolated by Geim and Novoselov for which they received the Nobel Prize for physics in 2018 [18]. Recently it has garnered a lot of attention due to its exceptional and distinctive physical, electrochemical, optical, mechanical properties. It is also termed as 'super carbon' for the same reasons. Hence it has been thoroughly studied and it's aid in areas such as environmental pollution control [19], Nano electronics, sensors [20], polymer composites [21], super capacitors [22], gas separation [23], and energy storage materials [24] have been reported. Graphene can be obtained by several methods some of which are chemical exfoliation, chemical vapor deposition, micromechanical cleavage of graphite, and epitaxial growth

1.2.1 PROPERTIES OF GRAPHENE

Graphene is a pure type of carbon in which each particle is accessible for concoction response from different sides. Particles at the edges of a graphene sheet have unique chemical reactivity. It has the most elevated proportion of edge atoms. Impurities inside a graphene sheet increase its substance reactivity. Its thermal conductivity and mechanical strength may coordinate the astounding in-plane characteristics for graphite (around $3,000 \text{ Wm}^{-1}\text{K}^{-1}$ and $1,060 \text{ GPa}$), independently their break quality should be proportionate to that of carbon nanotubes for practically identical sorts of defects, and progressing examinations have seemed individual graphene sheets have exceptional electronic transport properties.

Graphite, shoddy and available in colossal sum, tragically does not immediately shed to yield individual graphene sheets. Here we present a general procedure for the arranging of graphene-polymer composites by methods for complete shedding of graphite and sub-nuclear measurement dispersing of individual, falsely washed graphene bed covers inside polymer has. A polystyrene-graphene composite confined by this course shows a pervasion edge of around 0.1 volume percent for room-temperature electrical conductivity, the most insignificant uncovered a motivation for any carbon-based composite beside those including carbon nanotubes; at only 1 volume percent, this composite has a conductivity of generally 0.1 Sm^{-1} , sufficient for some electrical applications. Our base up mixture approach of tuning the graphene sheet properties gives a path to a far-reaching new class of graphene-based materials and their usage in a combination of employments.

1.3 GRAPHENE OXIDE

Graphene oxide (Henceforth referred to as GO) is basically functionalized graphene which has oxygen containing chemical groups such as carbonyl, carboxyl, epoxide, and hydroxyl groups. It is a carbon sheet that is one atom thick and made up of condensed six membered rings. It is obtained by the process of oxidation of graphene.

1.3.1 ADVANTAGE OF GRAPHENE OXIDE

Some of the major advantages of graphene oxide are:

- Possesses large theoretical specific surface area of up to $(400\text{--}1500 \text{ m}^2 \text{ g}^{-1})$. High surface area usually increases the reaction sites.
- Exhibits higher and efficient utilization of surface area because both sides of the nano sheets are accessible.

- Cost efficient and can be easily reduced back to its original form of graphene by chemical reduction processes or thermal annealing.
- Acts as an efficient support for metal nanoparticles and proves to exhibit fascinating catalytic properties. [25-26]
- Effortlessly dispersible in water and other organic solvents.

All of the above mentioned details are mere proof that GO is indeed an ideal candidate for a catalyst and hence it was conclusive to be suitable for our work.

1.3.2 APPLICATION OF GRAPHENE OXIDE

Its applications are extensive, a few of which are given below:

- Electronics: graphene based field effect transistor (GFET) hole transport layer in polymer solar cells and LEDs
- Energy storage: Lithium ion batteries super capacitors,
- Biomedical applications: Drug delivery systems,
- Biosensors: FRET sensors, Folic acid functionalized GO was used as a component in a system used to detect human cervical cancer and human breast cancer cells

Ever since its discovery, GO has proven to be a beacon that gave rise to many other major research work beneficial to humanity. The reason for its widespread application is its benefits which outweigh any shortcomings.

1.4 REDUCED GRAPHENE OXIDE

Reduced graphene oxide has similar mechanical, optoelectronic or conductive properties to pristine graphene because it possesses a heterogeneous structure comprised of a graphene-like basal plane that is additionally decorated with structural defects and populated with areas containing oxidized chemical groups. The graphene-like properties make reduced graphene oxide a highly desirable material to be used in a plethora of sensorial, biological, environmental or catalytic applications as well as optoelectronic and storage devices. To further advance the development of the existent technologies and to design novel and better applications based on reduced graphene oxide, it is first necessary to understand which synthetic routes and processing strategies are suitable to significantly boost specific properties of this material alone or as a component in various composites. Therefore, in this work, we review the most important categories of recent applications based on reduced graphene oxide, with the emphasis on the relationship between the enhanced composite/device functionality and methods used to synthesize, to functionalize and/or to process and to structure reduced graphene oxide[27]. Generally, this method relies on the use of chemical reagents such as hydrazine and its derivatives hydrazine hydrate and dimethyl hydrazine. In these cases, reduction of GO to RGO sheets is achieved through the use of hydrophobicity by simply adding these liquid reagents to a GO aqueous dispersion [28]. Other reagents including sodium borohydride (a good agent that efficiently reduces C=O groups) [29], a combination of sodium borohydride and concentrated sulfuric acid (reduction of GO is improved by an additional dehydration process)[30], hydroiodic acid [31] ascorbic acid,[32]etc. can also be used. Chemical reagents can be completely eliminated when employing the electrochemical method.

1.4.1 APPLICATION OF REDUCED GRAPHENE OXIDE (rGO)

rGO have an extremely high surface area; therefore, these materials are considered for usage as electrode materials in batteries and double-layered capacitors, as well as fuel cells and solar cells [33] Nanocomposites of GO/rGO can also be used for high-capacity energy storage in lithium ion batteries. In this case, electrically insulating metal oxide nanoparticles were adsorbed onto rGO to increase the performance of these materials in batteries [34].

GO/rGO is a fluorescent material that could be used for bio sensing applications, for early disease detection, and even for assisting in finding cures for cancer and detecting biologically relevant molecules. GO has been successfully used in fluorescent-based biosensors for the detection of DNA and proteins with a promise of better diagnostics [35] Closing these Nano capillaries using chemical reduction with hydro-iodic acid creates rGO films that are completely impermeable to gases, liquids, or strong chemicals .100 nm thick. Glassware or copper plates covered with such graphene “paint” can be used as containers for corrosive acids. Graphene-coated plastic films could be used in medical packaging to improve shelf life

1.4.2 ADVANTAGE OF REDUCED GRAPHE NE OXIDE (rGO)

- It is ultra-light and also immensely tough.
- It is thinnest material possible and it is completely transparent which can Transmit more than 90% of light.

- It can be used for wide variety of application such as flexible displays (OLEDs, LCDs, RAM, energy efficient transistors, energy storage devices, textile electrodes, copper nanowires, thermal management, spintronics etc.,
- It can be used in clothing which uses graphene based photo-voltaic cells as well as Super conductors. Due to this while in the pockets itself.

1.5 COPPER OXIDE NANOCOMPOSITES

Copper oxide (CuO) is a semi-conducting compound with a monoclinic structure. CuO has attracted particular attention because it is the simplest member of the family of copper compounds and exhibits a range of potentially useful physical properties, such as high temperature superconductivity, electron correlation effects, and spin dynamics. Copper oxide is relatively cheap, easily mixed with polarized liquids (i.e., water) and polymers, and relatively stable in terms of both chemical and physical properties. Highly ionic Nano particulate metal oxides, such as CuO, may be particularly valuable antimicrobial agents as they can be prepared with extremely high surface areas and unusual crystal morphologies.

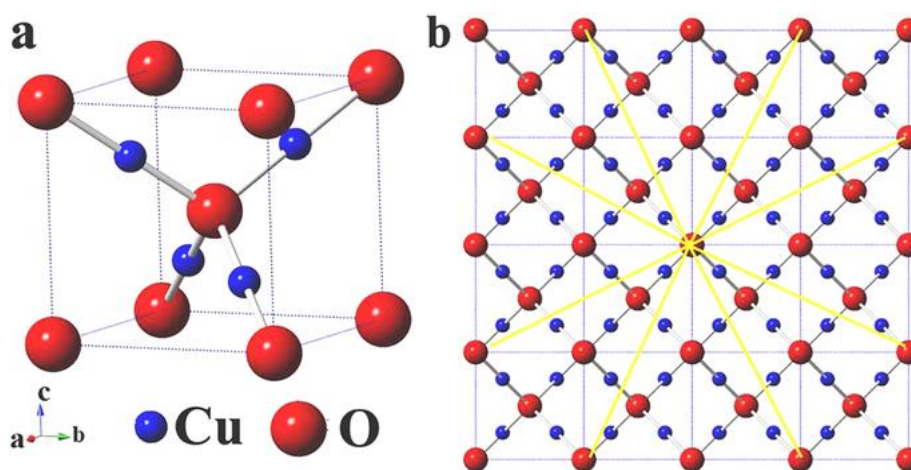


Figure 1.2 Crystalline structure of copper oxide Nanocomposite

1.6 rGO-COPPER OXIDE NANOCOMPOSITES

Integrating nanoparticles into GO enhances the properties and functionality of the nanocomposites due to the synergetic effects between GO Nano sheets and the nanoparticles. A facile and novel hydrothermal method of preparing copper oxide–reduced graphene oxide (CuO–rGO) nanocomposites has been developed.

In this method simultaneous formation of CuO and reduction of GO takes place without using any foreign materials. The synthesized composites were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive X-ray (EDX) spectroscopy. 4-Nitrophenol (4-NP) was used as a probe to assess the catalytic activities of the prepared CuO–Rgo [36].

1.7 SYNTHESIS OF rGO-CuO NANOCOMPOSITES

Initially, GO was first synthesized by Brodie in 1859, using graphite as a precursor, along with a mixture of potassium chlorate and concentrated nitric acid and the product obtained was entitled to be graphitic oxide. However, after the encounter and widespread research on it, it was then named as graphene oxide. In solid state, the GO powder is essentially graphitic oxide. But when GO is dispersed in solvents, due to the partial exfoliation by solvent molecules, graphene oxide is produced. Hence it is possible to say that a single atomic layer of graphite oxide is graphene oxide. Other common methods for the production of GO include the Staudenmeir method, Hoffman method and Tour method. Of all the various methods however, Hummer's method [37] is considered to be safer and more efficient owing to its simplicity. It was developed in 1957 by Hummers and Offeman in 1957 using graphite, Sulphuric acid, sodium nitrate and potassium permanganate.

It involves the powerful oxidation of graphite producing graphite oxide, and further dispersing it to obtain Graphene oxide. To this day, it is largely used for the bulk production of GO. Over time it has undergone some modifications [38]. The primary reason for its wide usage apart from the obvious fact of simplicity is that this method produces a large carbon to oxygen ratio when compared to the rest. Hence, the Hummer's method was chosen for the synthesis of GO for this project. Different ratios of rGO–CuO nanocomposites, i.e. 1:3, 1:2, 1:1, 2:1, 3:1, 4:1, 5:1 and 6:1 were prepared while keeping the total weight of composite mixture to 0.3 g. Each rGO–CuO Nanocomposite was coated on stainless steel (1 cm²) electrode using n-methylpyrrolidone along with poly (vinylidene fluoride) as binder. A symmetrical super capacitor cell was constructed using rGO–CuO nanocomposites-coated electrodes using polypropylene separator.

1.8 MODIFIED HUMMERS METHOD

Hummers' method is a chemical process that can be used to generate graphite oxide through the addition of potassium permanganate to a solution of graphite, sodium nitrate, and sulfuric acid. It is commonly used by engineering and lab technicians as a reliable method of producing quantities of graphite oxide [39]. In our present project we have modified the measurements of the compounds, used for the experiments.

CHAPTER 2

LITERATURE REVIEW

Reza Eivazadeh-Keihan, Reza taheri-ledari, meryan saidai mehrabed, had synthesized effective combination of rGO-CuO Nanomaterials using poly (P-Phenylenediamino). It characteristic is to improve super capacitor. GO is synthesized by modified hummers method and CuO nanomaterial is synthesized and effectively combined with pPD in the presence of APS. The Nanocomposite is structurally characterized by FTIR, SEM, TGA, CV, EIS, CCD techniques. [40]

Miao liu, zeyuwang, peng song, zhongxi yang, qiwang, has synthesised graphene and copper for high performance acetone sensing at room temperature. They reported an efficient approach for the production of ultrafine CuO nanoparticles-decorated three-dimensional (3D) hybrid aerogel by employing MXene (Ti₃C₂T_x) and reduced graphene oxide (rGO) nanosheets as cobuilding blocks. The structural and morphological characterized by UV, SEM, and FTIR techniques. [41]

Saima Siddique, Zain-ul-abdin, Muhammad waseen, Taiba naseem, they have prepared Photo-catalytic, and antimicrobial activities of rGO/CuO Nanocomposites by the reduction of GO to rGO
Using the step simple chemical method. A Nanocomposite was characteristic by EDX, SEM, XRD, UV-Vis, and FTIR techniques. [42]

A. Sridevi, B. Balraj, N. Senthilkumar has synthesised of rGO-CuO/Ag Ternary nanocomposites via hydrothermal apparatus for optoelectronics and Super capacitor Application. Ammonia is also used as reducing agent at room temperature. This characteristic has analyzed by SEM, TEM, EDAX, NC and CV techniques. [43]

Abayori Badatunde Alayande, M. Obida has done antimicrobial mechanisms of reduced graphene oxide-copper oxide Nano Composites Film. In the case of *Pseudomonas aeruginosa* (PA-01).

Its characterization has been analyzed through the EDX, XPS, SEM, TEM, and FT-IR. [44]

CHAPTER 3

AIM AND SCOPE OF THE PRESENT INVESTIGATION

The thesis mainly highlights the synthesis, characterization and specific application of reduced graphene oxide and copper oxide nanoparticles and it plays a vital role in several areas or research, such as catalysis, sensors, photo catalysis, drug delivery and many other properties reported in the literature. Copper is the cheap metal and shows a broad spectrum of catalytic activity starting from

C-C coupling to heterocycle synthesis. This thesis also includes the use of synthesized reduced graphene oxide and copper oxide nanoparticles in various applications, including in medical field.

From Graphite powder, Graphene has been synthesised to reduce graphene oxide. Later adding copper acetate and followed by hydrothermal process for 24 Hours. The sample is filtered and washed. This sample is characterized by various analyses such as XRD, SEM, FTIR, and UV techniques.

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS

All the chemicals used in this experiment were of analytical grade and is used without any further purification. Graphite fine powder (98% Extra pure) was purchased from Loba chemicals, Sodium Nitrate NaNO_3 (99%) was purchased from Sigma Aldrich, Sulphuric acid H_2SO_4 (98%) was purchased from Loba chemicals, Potassium Permanganate KMnO_4 (99.5%) was purchased from SRL, Hydrogen Peroxide (30% W/V H_2O_2) was purchased from Loba chemicals, Ethanol and Acetone were Purchased from SRL. All of these solutions were prepared using deionized water.

4.2 EXPERIMENTAL METHODS

Synthesis of reduced graphene oxide (rGO)

Reduced graphene oxide is synthesized by “Modified Hummer’s Method”. 2 grams of graphite powder and 2 grams of sodium nitrate were added to 90ml of 98% concentrated Sulphuric acid. The solution kept for stirring for 4 hours. Very slowly 12 grams of Potassium Permanganate was added and the solution kept stirring for 30-35 minutes under the ice bath. After slowly added 120ml of DI water in the solution. Raise the temperature of the stirrer to 100°C . Leave the solution to be stirred for 10 minutes. Check the temperature of the solution. The solution must be in 90°C and stop heating. But leave the stirrer to be continued. After 120 minutes, in a beaker measure 10ml of H_2O_2 and 40ml of deionized water mix well and pour in the stirring solution. Leave the solution to be stirred for 60 minutes. Off the stirrer and leave the solution at rest to settle down. Once the reaction was completed, the solution was centrifuged, and the supernatant given for characterization to confirm the formation of GO.

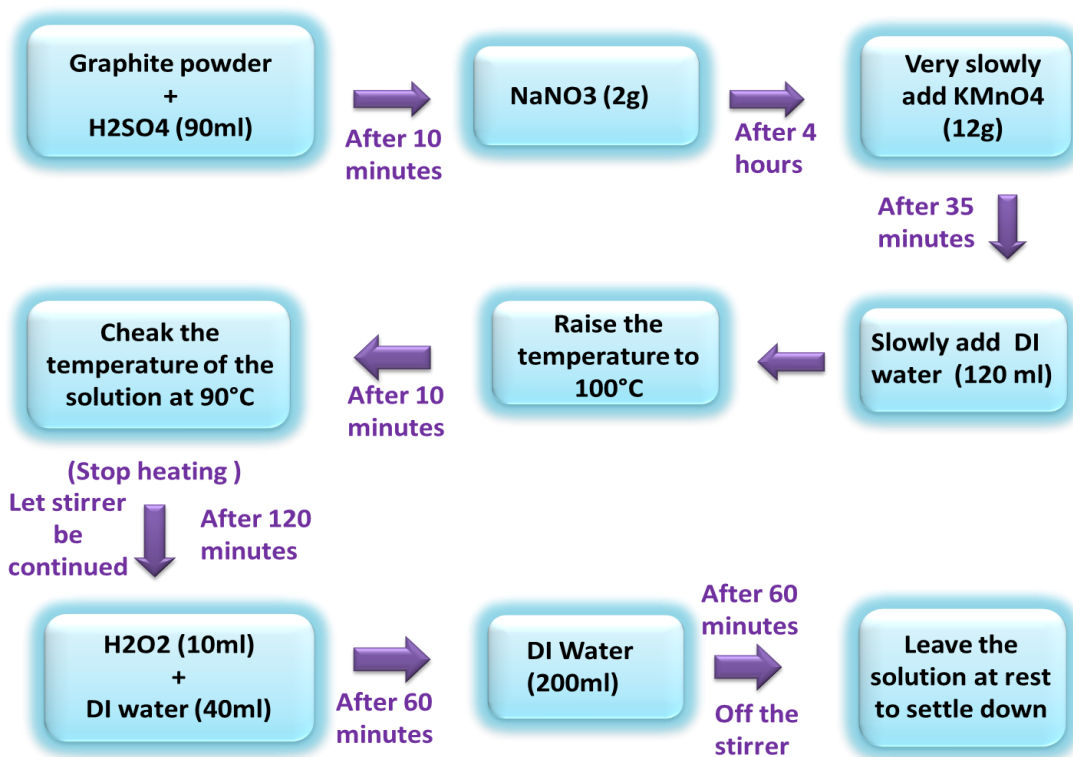


Figure 1.3 Synthesis of r-GO

Synthesis of rGO-CuO Nanocomposites

After the synthesis of reduced graphene oxide, in a beaker take 300 ml of dilute water and 200 ml of rGO and keep in the stirrer after 10 minutes soniheat the solution for 30 minutes and continue the stirrer. Separately Add 0.1 Mole of Copper acetate and NaOH of 0.1 mole is stirred for 60 minutes. Then this process is carried out by hydrothermal for 24 hours. Sample is filtered and washed. Thus the collected powder is rGO-CuO Nanocomposites.

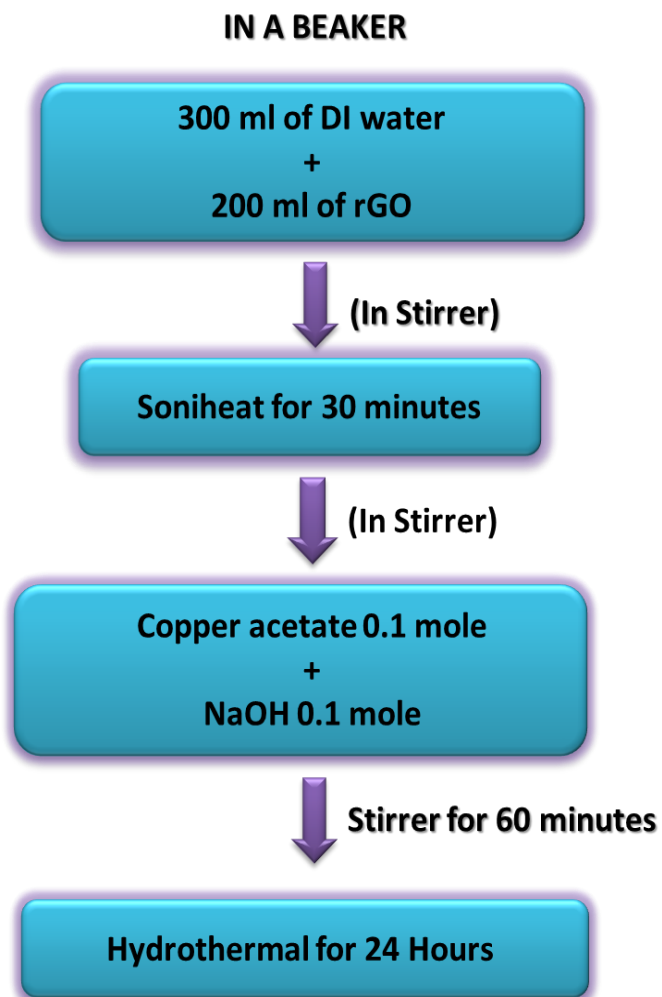


Figure 1.4 synthesis of rGO-CuO Nanocomposites

4.3 CHARACTERIATION TECHNIQUES

Crystal structure, Morphology, Chemical and structural feature, chemical bond, chemical composition, physical properties, band gap and adsorption, and application of synthesized nanoparticles were characterized using the following instruments. A Thesis includes X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), and Fourier Transform Infra-red Spectroscopy (FTIR). These highlighted techniques are presented in this project work. A brief information about the above techniques have been discussed in the following section

CHAPTER 5

RESULT AND DISCUSSION

Crystal structure, morphology, compositional information, organic functional groups on the surface and catalytic application of the synthesized nanoparticles were characterized using the following instruments. These include X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), Fourier Transform infra-red spectroscopy (FTIR) and ultraviolet spectroscopy (UV). Given below figure 1.5 rGO-CuO sample which is black in colour. These highlighted techniques are presented in this project work. Brief information about techniques has been discussed in the following section.



Figure 1.5 rGO-CuO Sample

5.1 XRD ANALYSIS OF rGO-CuO

When a material is irradiated with monochromatic light of wavelength around the inter atomic distance of the material, light gets dispersed by the atoms and interfere to give rise to diffracted light with a higher intensity. The arrangement of atoms in the material is related to the intensity of the scattered light.

If wavelength of light is λ and the incident angle is θ and the constructive interference is obtained at an angle 2θ from the incident beam of light if Bragg's equation is satisfied

Bragg's equation is given by,

$$n\lambda = 2d \sin \theta$$

Where, n is the diffraction order, d is the distance between the diffracting planes and θ is the Bragg's angle. Generally, in XRD Cu-K α is the incident light for which the $\lambda = 1.5405$ nm. Distance between the diffracted planes is calculated according to Bragg's equation and indexed by comparing it with the d spacing of the material from the joint Committee on Powder Diffraction Standards (JCPDS) data. rGO-CuO which exhibits several well-defined diffraction reflection peaks at $2\theta = 32.5, 35.01, 35.7, 38.8, 46.5, 48.8, 51.2, 53.3, 57.2$ and 62.1 corresponds to the (110), (002), (111), (-202), (020), (202), (-113), (-311), (220), (004), (22-2) crystal planes of CuO phase on rGO.

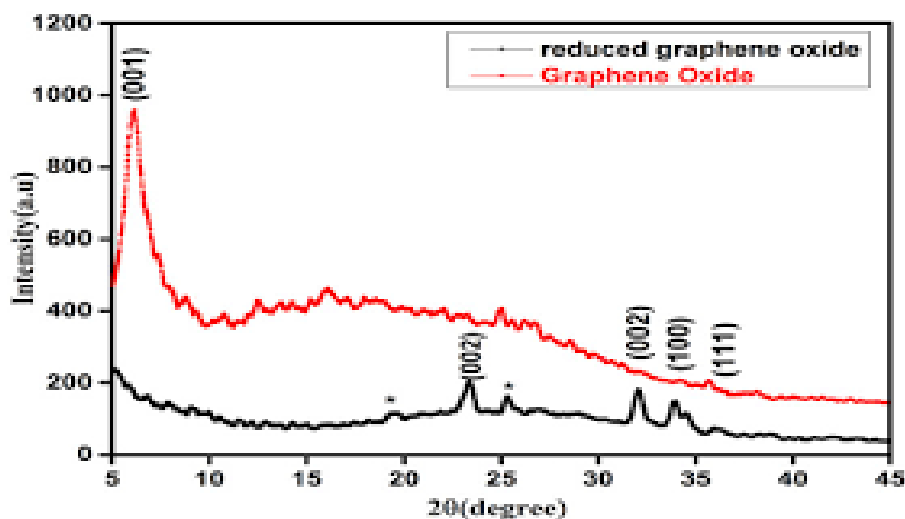


Figure 1.6 XRD peak for rGO-CuO

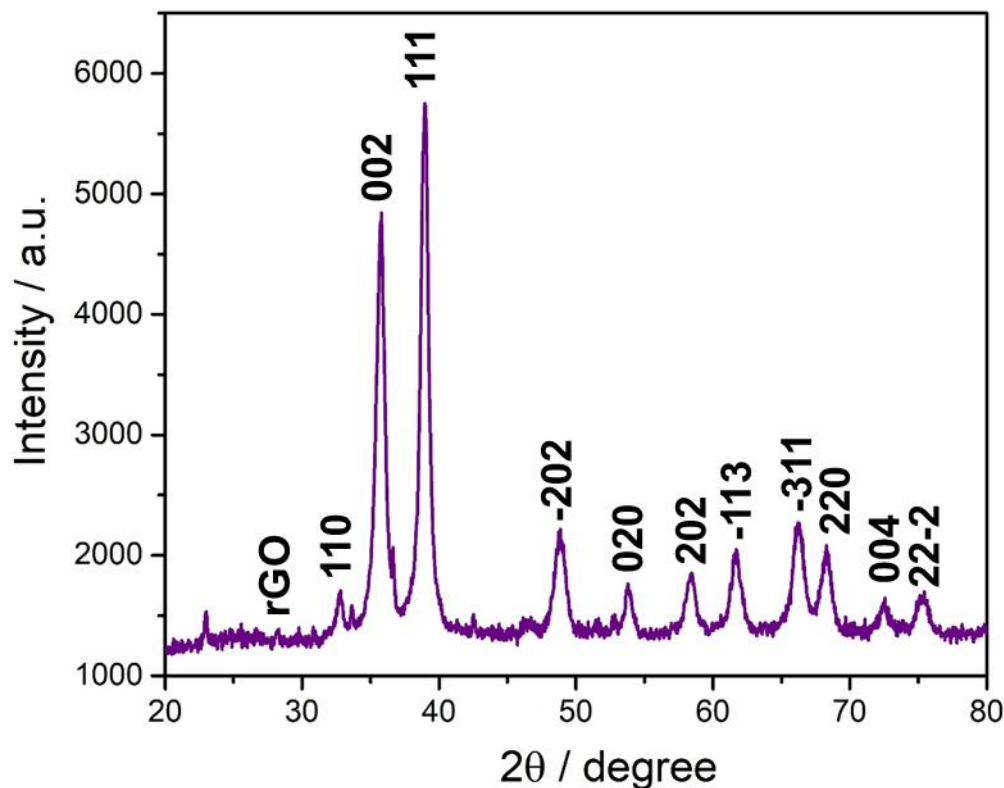


Figure 1.6 XRD peak for rGO-CuO

5.2 SCANNING ELECTRON MICROSCOPE (SEM)

High-resolution scanning electron microscope (SEM) is excellent for nanoparticle size and shape characterization, because the required sample preparation and image analysis is relatively quick and simple. [45] The scanning Electron Microscopy (SEM) image allowed a detailed study on the morphological structure of the nanoparticles. [46]

The Scanning electron microscope operates in the same manner as an optical microscope, but instead of detecting photon, it detects electrons scattering from the sample. The wavelength of electron can be accelerated by an electric potential. As a result, the SEM will magnify pictures up to 200,000 times [47].

The morphological variation in the synthesised sample nanoparticles were detected and analysed. rGO analysed by SEM is shown in figure 1.7(a) CuO doped on rGO thus synthesised at low temperature analysed and the images are displayed in Figure 1.7 (b) surface place of rGO.

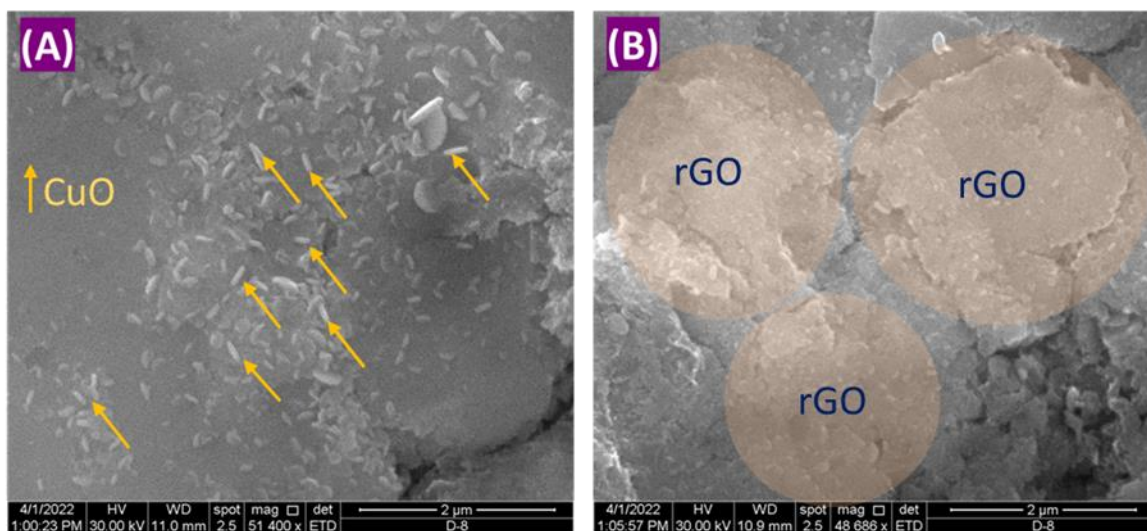


Figure 1.7 (a) Scanning Electron Microscopy (SEM) Image of rGO-CuO

(b) Scanning Electron Microscopy (SEM) Image of rGO

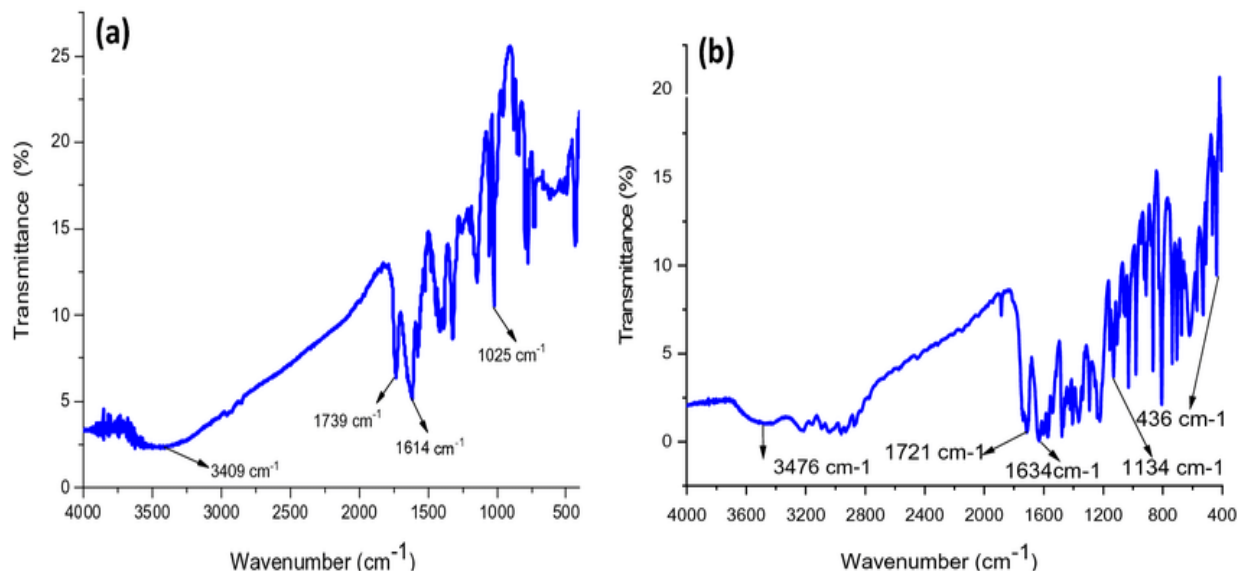
5.3 FOURIER TRANSFORMS INFRA-RED SPECTROSCOPY:

The FTIR analysis of reduced graphene oxide and Copper oxide is shown below in the figure 1.7 below. The formation of nanoparticles can be observed from its colour change. FT-IR spectroscopy was used to determine the presence of various functional groups in the prepared GO and rGO/ CuO Nanocomposite.

The O–H functional group vibrations and C–O stretching vibrations of the carboxyl group present at edges of the GO sheet appeared at 3409 cm^{-1} and 1721 cm^{-1} , respectively.

The adsorption peak at 1077 cm^{-1} was attributed to the C–O stretching vibrations, while C=C stretching vibrations in GO sheet appeared at 1614 cm^{-1} [48]. The absorption peak appeared at 3476 cm^{-1} was attributed to O–H stretching vibrations, which was significantly reduced in the rGO [49]. This decline in the intensity of the peak confirmed the reduction of the prepared GO into rGO.

The C–O stretching vibrations appeared at 1721 cm^{-1} . The peak appeared at 1634 cm^{-1} was attributed to C=C skeletal vibrations. Two peaks appeared at 821 cm^{-1} and 1478 cm^{-1} were credited to the Cu–OH stretching vibrations and epoxide stretching vibrations, respectively. These results support the successful preparation of the rGO/CuO Nanocomposite [50]. The absorption peak appeared at 436 cm^{-1} was attributed to Cu–O stretching vibrations which confirmed that CuO was completely decorated on rGO sheet [51]. The FT-IR spectra of the prepared GO and the prepared rGO/ CuO Nanocomposite are presented in Fig. 1.8



**Figure: 1.8 FT-IR spectra of (a) The prepared GO and
(b) The prepared rGO/CuO Nanocomposite**

5.4 UV-VISIBLE SPECTROSCOPY OF rGO-CuO

In case of GO, an absorption peak appeared at 232 nm, which was attributed to aromatic double bond and around 310 nm a peak appeared that was attributed to $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ transitions for C=O absorption [52]. In case of GO, the peak appeared at 231 nm and a small peak appeared due to $\pi \rightarrow \pi^*$ transition at 304 nm [53]. UV-Vis spectrum of the prepared GO is shown in Fig. 1.9

For the prepared rGO/CuO Nanocomposite, absorbance maximum appeared at 300 nm, which was attributed to $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ transitions and these transitions occurred because of aromatic carbon-carbon bond and carbonyl group presence [54]. The appearance of the absorption peak at 255 nm indicated the reduction of GO and replacement of OH and COOH group with CuO. It has been reported that red shift corresponds to the reduction of GO due to functionalization.

The greater red shift indicated the complete reduction of GO [55]. In literature, similar results have been reported for ZnS-rGO and TiO₂-rGO nanocomposites. The narrow band gap of CuO in the prepared rGO/CuO Nanocomposite was might be due to the greater carbon contents. The complete disappearance of the absorption peak at 300 nm, confirmed successful reduction of GO and anchoring of CuO on GO sheet [56]. Figure 1.9 shows the UV-Vis spectrum of synthesized rGO/CuO.

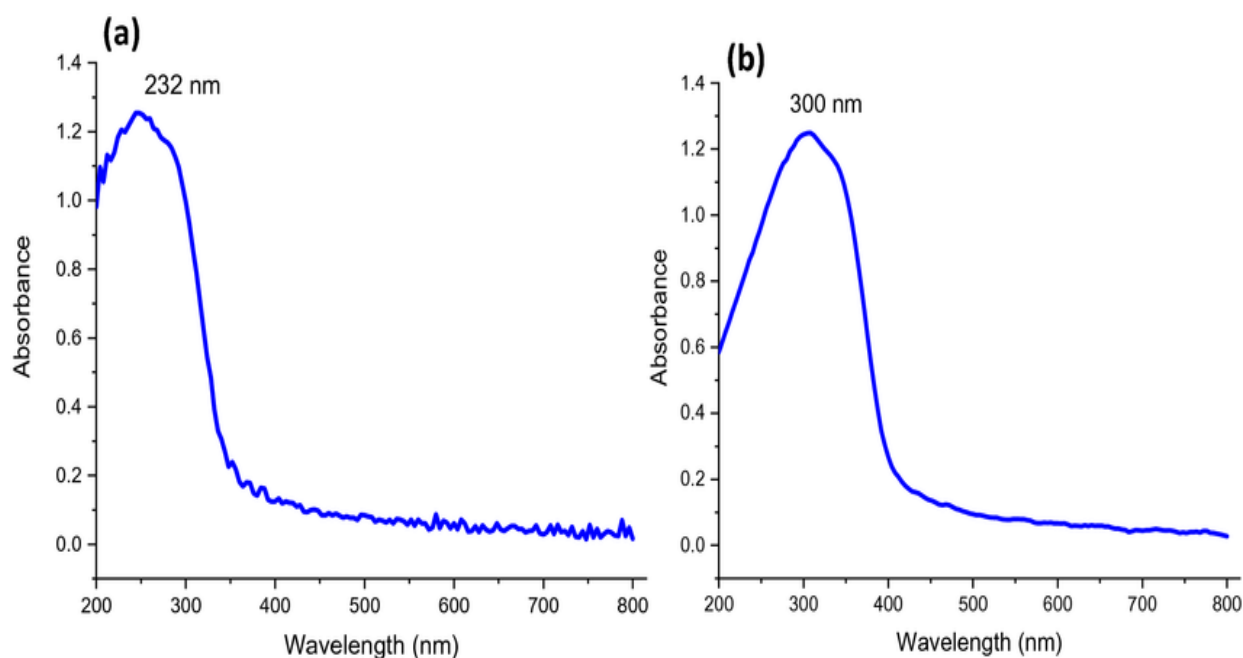


Figure 1.9 UV-Vis spectra of (a) The GO and (b) The rGO/CuO Nanocomposite

CHAPTER 6

SUMMARY AND CONCLUSION

A cost effective and simple technique was used for the synthesis of rGO/CuO Nanocomposite. The external morphology of the synthesized GO and the rGO/CuO Nanocomposite was investigated using SEM. The GO formed fakes-like structure. In case of the rGO/CuO Nanocomposite, CuO nanoparticles were anchored on the fakes-like structure of GO. It was found that many stacked and crumpled fakes like structures are present in the prepared Nanocomposite. The anchoring of CuO nanoparticles was done due to the development of interactions between CuO and the functional groups present on the surface of the GO. XRD patterns confirmed the successful synthesis of the Nanocomposite. It was concluded that the diffraction peak at 11.03° corresponds to GO (002) plane. Furthermore, three peaks of the rGO/CuO Nanocomposite appeared at 25.65° , 36.14° , 43.09° , 49.91° , and 69.52° , were attributed to Cu (111), Cu (200) and Cu (220). The appearance of band at 524 cm^{-1} due to Cu–O vibrations in FT-IR spectrum supported the successful preparation of the rGO/CuO nanocomposite. The prepared rGO/CuO nanocomposite was used for the catalytic conversion of 4-NP into 4-AP and photo-catalytic degradation of MB. It was found that the rGO/CuO nanocomposite is an excellent catalyst for the reduction of 4-NP to 4-AP due to the increased efficient charge transfer, light absorption and enhanced adsorption on the surface of composite due to the introduction of rGO. On the basis of results obtained it was concluded that the rGO/CuO nanocomposite exhibit high potential for the catalytic activity, It is also used for the defense field and solar cell, batteries and cellular imaging. The rGO/CuO nanocomposite is a non-toxic, environmental friendly and can be used for the removal of the pollutants from the contaminated water.

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