PHOTOTHERMAL AND ANTIBACTERIAL STUDY OF $Cu/Cu_2O/CuO$ NANOCOMPOSITE

PROJECT REPORT

Submitted by

AKHILA GEORGE

Register No:AM22PHY002

Under the guidance of

Ms. MINU PIUS

Assistant Professor
Department of Physics
St. Teresa's College (Autonomous), Ernakulam
Kochi – 682011

Submitted to

Mahatma Gandhi University, Kottayam

In partial fulfillment of the requirements for the award of the degree of

MASTER OF SCIENCE IN PHYSICS

DEPARTMENT OF PHYSICS

ST. TERESA'S COLLEGE (AUTONOMOUS), ERNAKULAM

DEPARTMENT OF PHYSICS

ST. TERESA'S COLLEGE (AUTONOMOUS), ERNAKULAM

CERTIFICATE

This is to certify that the project report entitled "**PHOTOTHERMAL AND ANTIBACTERIAL STUDY OF Cu/Cu_2O/CuO NANOCOMPOSITE**" is an authentic work done by **AKHILA GEORGE (AM22PHY002)** under my guidance at Department of Physics, St. Teresa's College (Autonomous), Ernakulam for the partial fulfillment of the requirements for the award of the Degree of Master of Science in Physics during the year 2023-24. The work presented in this dissertation has not been submitted for any other degree in this or any other university.

Supervising Guide
Ms. MINU PIUS

Head of the Department
Dr. PRIYA PARVATHI AMEENA JOSE

Place: Ernakulam
Date: 18-04-2024

DEPARTMENT OF PHYSICS

ST. TERESA'S COLLEGE (AUTONOMOUS), ERNAKULAM

M.Sc. PHYSICS

PROJECT REPORT

Name : **AKHILA GEORGE**

Register No. : **AM22PHY002**

Year of Work : **2023-2024**

This is to certify that the project entitled "**PHOTOTHERMAL AND ANTIBACTERIAL STUDY OF Cu/Cu_2O/CuO NANOCOMPOSITE**" is an authentic work done by **AKHILA GEORGE**.

Staff member in-charge

Ms MINU PIUS
Assistant Professor

Head of the Department

Dr. PRIYA PARVATHI AMEENA JOSE
Assistant Professor

Submitted for the university examination held at St. Teresa's College (Autonomous), Ernakulam.

DATE: 22/4/2024

EXAMINERS:

1. Dr. Issac Paul, 29/4/24
2. Dr. Gishamol Mathew, 22/4/24

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Author Name	N SORNAMUHI , AKHILA GEORGE
Course of Study	M.Sc. PHYSICS
Name of Guide	Ms. MINU PIUS
Department	Physics & Centre For Research
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Similarity	0% AI 15%
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DECLARATION

I, **AKHILA GEORGE,** final year MSc. Physics students of the Department of Physics and Centre for Research, St. Teresa's College (Autonomous), Ernakulam, do hereby declare that the project report entitled "**PHOTOTHERMAL AND ANTIBACTERIAL STUDY OF Cu/Cu_2O/CuO NANOCOMPOSITE**" has been originally carried out under the guidance and supervision of **Ms. MINU PIUS,** Assistant Professor, Department of Physics, St. Teresa's College (Autonomous), Ernakulam in partial fulfilment for the award of the Degree of Master of Physics. I further declare that this project is not partially or wholly submitted for any other purpose and the data included in this project is collected from various sources and are true to the best of my knowledge.

PLACE: ERNAKULAM

DATE:

AKHILA GEORGE

ACKNOWLEDGEMENT

I am deeply indebted to my project guide, **Ms. Minu Pius**, Assistant Professor, Department of Physics and Center for Research, St. Teresa's College Ernakulam for providing me an opportunity to carry out this project under her supervision. Her learned counsel, critical analysis of work, constructive comments, constant encouragement and understanding throughout the period of my project work have enabled me to complete this work. Frequent discussion with her at different stages of the work has served as unfailing source of intellectual stimulation to the occasional slackness in the mind.

I express my sincere gratitude to **Dr. Santhi A**, Assistant Professor, Department of Physics, St. Teresa's College Ernakulam for her constant support throughout the project by sharing different methodologies to carry out my project.

I place on record my sincere gratitude to **Dr. Priya Parvathi Ameena Jose**, Head of Department of Physics, St. Teresa's College Ernakulam for giving me permission and moral support for the successful completion of my project.

I acknowledge my thanks to Ms. Merin Joby, Research Scholar at the Department of Physics and Center for Research, St. Teresa's College Ernakulam for extending her support whenever sought.

I extend my sincere thanks to the faculty members of Botany and Zoology Departments for providing me their support with the instrumentation part in my project.

I thank all my teachers and non-teaching staff of the Physics Department for their moral support given to me in carrying out my project.

Most importantly, I express my sincere gratitude to my parents and friends whose unwavering support has helped me throughout in completing my project.

PHOTOTHERMAL AND ANTIBACTERIAL STUDY OF $Cu/Cu_2O/CuO$ NANOCOMPOSITE

CONTENTS

ABSTRACT

This work is in the realm of nanotechnology, and investigates the thermo-optic and antibacterial properties of $Cu/Cu_2O/CuO$ nanocomposite. $Cu/Cu_2O/CuO$ nanocomposite was prepared through electrolysis method and was characterized using X-ray Diffraction, Scanning Electron Microscopy, UV-Visible spectroscopy and photoluminescence spectroscopy. Thermal diffusivity of prepared sample was investigated using dual beam thermal lens technique, and the thermal diffusivity was studied for different concentration of the sample of nanofluid. Antibacterial efficacy of the sample was carried out against gram-negative and gram-positive bacteria E. coli and S. aureus respectively.

CHAPTER 1
INTRODUCTION

1.1 Nanotechnology

Richard Feynman in his talk at the American Physical society in 1959 stated that "There is plenty of room at the bottom". This was the emergence of a new idea of nanoscience. Norio Taniguchi was the first to use the term 'Nanotechnology'. Nanotechnology is the creation and manipulation of materials on the scale of atoms and molecules. Materials that are created in this scale typically exhibit unique physical, chemical, and biological properties. This is due to quantum mechanical effects. They have large strength and conductivity due to their small size. They also have large surface to volume ratio, which makes them useful in various fields. Nanomaterials show various optical and electronic properties which makes them useful in applications like sensors, displays, and photovoltaics. Though the usage and manipulation of these nanosized particles were known, but its visibility was a challenge. In 1980's the invention of STM was breakthrough in the field of nanotechnology. This played an important role in the discovery of fullerenes and carbon nanotubes in later stages.

There are two approaches through which we can synthesis nanomaterials.

Bottom-up approaches-

In this approach we can synthesis nanomaterials from atoms, molecules or from nanoparticles. This is done through techniques like molecular beam epitaxy, chemical vapour deposition, atomic layer deposition, sol-gel technique etc.

Top-down approaches-

In this approach we can synthesis nanomaterial by breaking bulk materials. This is done through techniques like ball milling, lithography, etching etc.

Comparing both these approaches bottom-up approaches have several advantages over top-down technique because we can obtain materials with perfect edges through bottom-up approach. Also bottom-up approach have more control over the material size and less material is wasted in bottom-up products.

Nanoparticles can be classified into organic and inorganic based on the composition of molecule with which they are made of.

Organic nanoparticles –

Nanoparticles that are composed of carbon-based molecules are called as organic nanoparticles. Some of the examples are micelles, liposomes, dendrimers, ferritin and polymers etc. One of the main advantage of organic nanoparticle is that they can be broken down naturally. Organic nanoparticles are extremely sensitive to light and heat. Therefore they can be used for the transportation of pharmaceuticals. They are most importantly used to transport medications to the desired locations. Some of the organic nanoparticle such as micelles and liposomes are said to be non-toxic and biodegradable. Organic nanoparticles are usually observed in the form of Nano capsules or nanospheres.

Inorganic nanoparticles –

Inorganic nanoparticles are not composed of carbon-based molecules. Compared to organic nanoparticles, they are hydrophilic, non-toxic and highly stable. They are again subdivided into metal based and metal oxide-based nanoparticles.

1.2 Metallic nanoparticles

Nanoparticles that are synthesized from metals are called as metallic nanoparticles. Almost every metal can be transformed into its corresponding nanoparticles. Some of the examples are gold, silver, platinum, copper, cobalt, zinc etc. Metallic nanoparticles exhibit unique surface plasmon resonance and optical properties and this has created a new pathway for the nanotechnology. For example, gold nanoparticle appears as a red colored when size is 20nm and when the size is around 200nm they are found in bluish color. Metallic nanoparticles have gained much attention to researchers in various fields of science and technology. Especially the noble metal nanoparticles. Main advantage of metallic nanoparticles is that they can interact with other particles. This is because of their large surface to volume ratio. Metallic nanoparticles can be used to treat effected tissues and cells without effecting and damaging other cells and tissues. Silver and gold nanoparticles do show antibacterial effect to both gram positive and gram negative bacteria. They exhibit large zone of inhibition. Even though other metallic nanoparticles like copper and zinc show antibacterial properties, they show small

zone of inhibition compared to gold and silver nanoparticles. Characteristics of metallic nanoparticles are strongly dependent on various features like experimental conditions, stabilizing agent used, concentration and type of stabilizing agent etc. Metallic nanoparticles have several advantages like enhancing Rayleigh scattering, surface enhanced Raman scattering, strong plasma absorption, biological system imaging. They also have many disadvantages. One of the main problem is the stability of metallic nanoparticles. It is extremely difficult to stabilize them. They can easily get transformed into oxide form even if we use stabilizing agent. For example copper nanoparticles. They can easily get oxidized into CuO and Cu_2O. Due to this reason it is also possible to observe metallic nanoparticles as a mixture of metallic and metal oxide nanoparticles.

1.3 Copper nanoparticles

Copper is a chemical element with symbol Cu. It has an atomic number 29. It is a malleable and ductile metal with appreciable electrical and thermal conductivity. Copper nanoparticles have appeared as diversely usable new material with great potential. Compared to other metals copper has a better capacity for catalysis, electricity, and antimicrobial applications. This property is because of the good surface area to volume ratio and quantum size effects. Copper nanoparticles can be synthesized in a number of ways, including chemical reduction, thermal decomposition, and electrochemical methods. They have a wide range of applications from catalysis to environmental cleanup in various field. Though it has many viable application, one major disadvantage of copper nanoparticles is that it can easily get oxidized and convert to CuO and Cu_2O forms. To minimize this discrepancy there were many agents called the stabilizing agents, some of them are- PVP, ascorbic acid, starch etc. Copper is an important element all living organisms, especially in the case of humans. They are required for the functioning of different metabolic activities. They are also involved in development of red blood cells and also helps the immune system to fight against infections and injured tissues. Copper nanoparticles have very good photothermal conversion efficiency and antigen capturing property. Nanoparticles of copper possess high surface area to volume ratio due to their minute size; thus they are highly reactive making them suitable in catalytic applications. Though many metal nanoparticles exhibit anti-microbial activities, copper nanoparticles show evident antimicrobial activity due to its ability to release copper ions. This makes it highly active in many pathogens. On the other hand, copper nanoparticles can be used for applications like, electronics and sensors due to its high conductive nature. In visible spectrums, copper nanoparticles like many other noble metals such gold and silver exhibit surface plasmon resonance. However, compared to other metals

the surface plasmon resonance of copper nanoparticles can be tuned. With many advantages on one side, copper nanoparticles are found to be toxic at higher concentrations. Their toxicity is size dependent. Compared to zinc oxide and gold nanoparticles, antibacterial activity of copper nanoparticle is less as they are required in large amounts to show the same result.

1.4 Different synthesis methods of copper nanoparticles

1.4.1. Thermal decomposition

This method involves dissolving copper chloride and sodium oleate in a mixture of hexane, ethanol and distilled water. This is then heated and was transferred to a separation funnel to eliminate the aqueous residues. The organic phase that contain copper oleate complex and hexane is then washed with distilled water. This complex is then transferred to petri dish to allow evaporation to remove residue solvent. Next copper oleate complex was mixed with oleic acid and phenyl ether at room temperature. This is heated at 250°c for 30 minutes. During the heating process, the solution will turn brown in color indicating the presence of copper nanoparticles. This solution is then cooled at room temperature and is washed with ethanol for multiple times to eliminate any residues present. The finally obtained nanoparticles are collected through the process of centrifugation.

1.4.2. Chemical reduction method (using copper nitrate, PVP and hydrazine hydrate).

This method involves the addition of polyvinylpyrrolidone in water. Then heat it is to 60°c to dissolve it properly. Then add copper nitrate in to it and stir it. Final step of this method is addition of hydrazine hydrate in to it by drop by drop until color of the solution changes to green which indicates the formation of copper nanoparticle in the solution. There are many factors that can affect the synthesis such as concentration of PVP, concentration of hydrazine hydrate, concentration of copper nitrate and temperature. PVP is used as a capping agent so if we increase its concentration size of the particle will be small. Precursor of this process is copper nitrate, so higher the concentration higher will be the size copper nanoparticle. Hydrazine hydrate is reducing agent so increase its concentration can lead to agglomeration of particle. One of the main factor of this method is temperature.

1.4.3. Green synthesis method

In this method leaves of J. curcas is needed. Extract of these leaves are used for the synthesis. After washing the leaves with distilled water, cut it in to small sizes. This is again washed with distilled water. Dry these pieces under the presence of sunlight. These leaves are then incubated at 40°c to 60°c after immersing it in distilled water. This is done to prepare plant extract. Next step of this method is preparation of 80mL of copper chloride. 20mL of plant extract is added to the 80mL of copper chloride. Stir it well for 24 hours at room temperature. We can observe the change in solution from deep brown to yellowish brown. This indicates the presence of copper nanoparticles. We can also perform green synthesis of copper nanoparticle using neem leaves.

1.4.4. One pot synthesis

In this method sodium borohydride is used as the reducing agent. This method include preparation of copper ammonia complex solution. This is prepared by adding 1g of copper metal in 10mL of ammonia solution. This leads to the formation of blue copper ammonia complex. Then add hydrochloric acid in to it, mix it well till solution become neutral. After this step add 100ml of 0.25 M $NaBH_4$ that contain starch as stabilizing agent drop by drop in to the solution for half an hour. The color of the solution will change to dark brown indicating the presence of copper nanoparticles. Centrifuge the solution to separate nanomaterial from solution.

1.4.5. Chemical reduction process (using copper sulfate pentahydrate as precursor salt and starch as capping agent).

This procedure include preparation of ascorbic acid solution in deionized water. In to this separately prepared 0.01 M solution of copper sulphate that is made in deionized water is added by magnetic stirring. 1M solution of NaOH that is also made in deionized water is added in order to adjust the pH. This solution is then stirred for 30 minutes. After this 0.1M solution of $NaBH_4$ that is also prepared in deionized water is added in to it. Continue stirring until blue color solution change to red brown color. This indicate the presence of copper nanoparticle.

Even though these synthesis methods are for copper nanoparticles, there are high possibilities to observe the presence of copper oxide nanoparticles along with copper nanoparticles.

1.5 $Cu/Cu_2O/CuO$ Nanocomposite

One of the main problem of copper nanoparticles is its instability. It is extremely difficult to stabilize them. They can easily get oxidized into CuO and Cu_2O. Majority of time instead of getting copper nanoparticle alone we would be getting copper and copper oxide nanoparticle mixture. Such a mixture of copper, Cu_2O and CuO nanoparticle is called as $Cu/Cu_2O/CuO$ nanocomposite. They also have properties similar to that of pure copper nanoparticles. They exhibit high conductivity and have high surface to volume ratio. $Cu/Cu_2O/CuO$ nanocomposite can be prepared directly through many methods such as green synthesis method, electrodeposition method, electrochemical method, wet chemical method, etc. One of the main advantage of such a nanocomposite is that they show large antibacterial activity as well as catalytic activity compared to pure copper nanoparticle.

$Cu/Cu_2O/CuO$ nanocomposite has various electronic and optoelectronic applications due to their electronic properties. Some of these include field emission uses, solar cells, FETs, electrode for lithium ion batteries, etc. They are also used in antimicrobial applications, chemical sensors, magnetic storage media, etc.

1.6 Nanofluids

Nanofluids are a new class of fluids which is made by suspending nano-sized materials like nanodots, nanowires, nanotubes, nanosheets, etc in a base fluid. The addition of nanoparticles in the fluid can increase the thermal conductivity of the fluid and provides a way to increase their heat transfer properties. Water is usually used as a working fluid because of its extensive availability. But due to low thermal conductivity, they are not considered as an adequate heat transporter. Ethylene glycol and engine oil, in certain applications, are used as heat transfer fluids. Even though they have high thermal conductivity compared to water due to their toxic nature their usage is limited. Due to unsatisfactory thermal properties of conventional fluids, it was necessary to find a new class of fluids with high thermal conductivity. That’s how nanofluids emerged.

Nanofluids have a unique thermal transport phenomenon that show evident improvement in thermal properties such as thermal conductivity, thermal

diffusivity, viscosity and density in contrast to base fluids. Compared to conventional base fluids, they are a better alternative to save energy. Specific heat is an important property of a material to specify its thermal properties. In the case of nanofluids it was found that specific heat can vary depending on size and concentration of the nanoparticles, concentration of base fluids, and the type of base fluids used. Specific heat of the nanomaterials used in the base fluids decrease with increase in volume fraction.

Two methods are usually carried out for the preparation of nanofluids. They are the one-step and two-step methods respectively.

Two-step method-

In two-step method, nanoparticles are first synthesized through chemical, physical or biological method. Then the prepared nanoparticles are dispersed in to the base fluids. This is done with the help of intensive magnetic force agitation, ball milling, high-shear mixing and ultrasonic agitation. Through this method, nanofluids can be produced in large scale. One of the main disadvantage of two step method is aggregation of nanoparticles in nanofluid. Though surfactants can be added to improve the stability, it is still difficult to maintain the stability in high temperature applications.

One-step method-

In one-step method preparation of nanoparticles and dispersion of them in nanofluids are done simultaneously. In comparison with the two-step method, nanofluids synthesized through one-step is more stable. The stability is more in this this case as the steps like drying, storage, transportation is not needed here. Due to this, chances of agglomeration of nanoparticles in nanofluids prepared through one-step method is less. One of the main disadvantages of one-step method is that, this method is expensive to be performed. And the large-scale preparation of nanofluids through this method is not possible.

Nanofluids are mainly classified into two; single material nanofluids and hybrid nanofluids. In single material nanofluids, only one type of nanoparticles will be present in the nanofluid whereas hybrid nanofluids will have more than one type of nanoparticles suspended in the base fluid.

Nanofluids have a wide range of applications. They are used in cancer therapies, electronics, solar cells, drug delivery, nano cryosurgery, heat exchangers, refrigeration, automobile, etc.

1.7. REVIEW OF LITERATURE:

Various studies have been carried out by the researchers on the synthesis of copper nano particles and their characterization, thermal diffusivity and antibacterial properties. The work on Nano sized copper particles (Theivasanthi and Alagar 2011) by electrolytic synthesis and characterizations revealed that the synthesis rate is much faster than other methods and this approach is suitable for large scale production. XRD studies revealed a high degree of crystallinity and monophasic nature of copper nanoparticles. Microbiology essay proved that copper nanoparticles have antibacterial activities and are found effective against Escherichia coli and Bacillus megaterium bacteria. Harshavardhan (2014) had reported the research work on synthesis and characterization of copper nanoparticles by electrochemical method. In his studies, the effect of pH on copper nanoparticles synthesis has been done at different pH levels. Mladen Franko and Chieu D. Tran (2010) have reported on the instrumentation and unique properties and capabilities of Thermal Lens Techniques for studying the photothermal property of the synthesized nano particles.

CHAPTER 2
EXPERIMENTAL METHODS

2.1. Synthesis of copper nanoparticles

There are many physical, chemical and biological methods through which we can synthesis copper nanoparticle. Electrolysis is a method that uses direct current to perform a chemical reaction or we can say it is method of conversion of electrical energy to chemical energy. It is a chemical method which do have many advantages compared to other methods. It is an environmentally friendly method because it doesn't involve any toxic chemicals. Main advantage of this method is purity of nanoparticles. It is the best method that helps in controlling particle size by controlling factors like electrolysis concentration, time taken for procedure, voltage and current supplied etc. Overall it is an exceptional method because comparatively it requires less time.

Copper nanoparticles are synthesized through an electrolysis method using copper sulfate pentahydrate ($CuSO_4.5H_2O$) as electrolyte and copper plates as electrodes.

2.1.1. Preparation of 1% concentration of copper sulfate solution

To create 1 percentage of copper sulfate solution we added 4g of copper sulfate pentahydrate in 400ml of distilled water. We can create it through following steps

- Take 400ml of distilled water in a well cleaned beaker.
- Take 4g of copper sulfate pentahydrate after weighing it with weighing balance.
- Add this to the beaker that contains 400ml of distilled water.
- Stir it continuously until we obtain a well dissolved solution.

2.1.2. Cleaning of electrodes used for electrolysis

It is necessary to make sure that the electrodes that we use is well cleaned before using it. We can do it through following steps

- Prepare a cleaning solution by mixing equal amounts of acetic acid and hydrogen peroxide. This helps in removing any oxides or dirt present in it.
- Immerse the copper plates in this solution (completely immersed) for 30 minutes.
- After that scrub the plates using soft brushes.
- After scrubbing, wash it properly with water. This helps to remove remaining contaminants on the surface.
- After drying the electrodes, place it in a clean environment to prevent recontamination.

2.1.3. Experimental setup

Basic electrolysis setup consists of few components such as electrolysis cell, electrodes, power supply etc.

- Electrolysis cell
  It consists of a container filled with an electrolyte solution and two electrodes immersed in it. This is where the electrolysis reaction takes place.
- Electrolyte solution
  Electrolyte solution is a solution that can conduct electricity. They Commonly contain salts and bases.
- Power supply
  DC power supply is needed to provide electrical energy for the process.

- Anode and cathode
  These are electrodes used in electrolysis. Anode is the positively charged electrode where the oxidation takes place. Cathode is negatively charged electrode where the reduction takes place.

- Wires and connectors
  Wires are required to connect electrodes to power supply. Make sure that there is good electrical contact between them.

In our set up electrolyte used is prepared 400 ml of copper sulfate solution and electrode is copper plates.
Immerse the well cleaned copper plates into prepared copper sulfate solution. Make sure that they don't touch each other. Connect one plate to the positive terminal of the power supply and one to the negative terminal using wires and connectors. Plate connected to positive terminal is anode and the one connected to negative terminal is cathode.

Fig 2.1 electroplates immersed in copper sulfate solution and connected to power supply.

2.1.4. Procedure

12V power supply is given to the electrodes. After sometime it was observed that there was some brown coloured deposition on the cathode. These brown colored substance is the required copper nanoparticles.

When a steady current is passed through the electrolytic cell, copper sulfate solution gets dissociated due to ionization. Copper ions will be removed from anode and will settle on cathode. Electrons flow from negative to positive terminal causing Cu^{2+} to move towards cathode and SO_4^{2-} to move towards anode.

$$CuSO_4 \longrightarrow Cu^{2+} + SO_4^{2-}$$
$$Cu + SO^{2-}{}_4 \longrightarrow Cu^{2+} + SO_4^{2-} + 2e^-$$
$$Cu^{2+} + 2e^- \longrightarrow Cu$$

Nanoparticles are carefully collected into a petri dish. This collected substance is washed multiple times using distilled water. This solution is filtered to remove the and it is kept to dry.

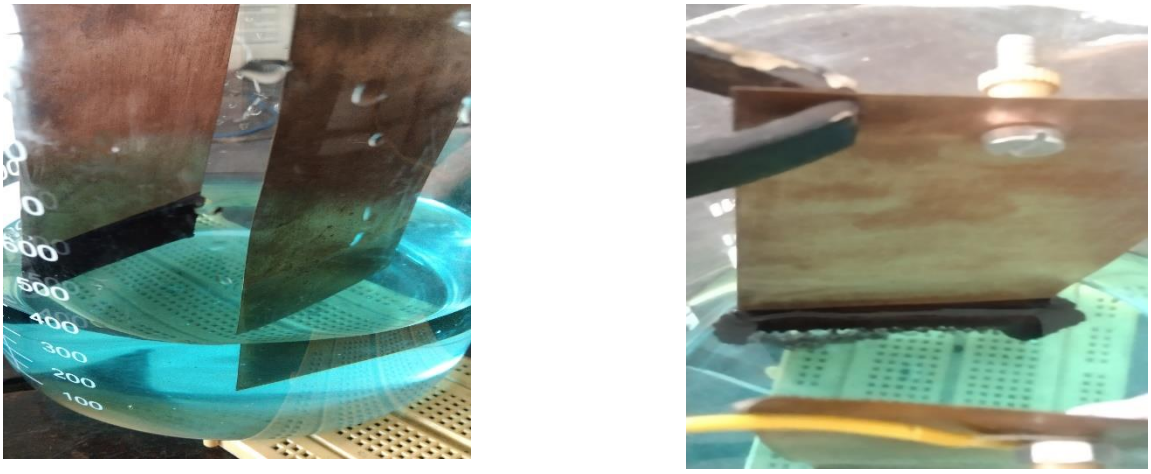

Fig 2.2. Copper nanoparticles getting deposited on cathode

Size of the nanoparticles depend on the voltage given, reaction time, electrolyte concentration.

- Voltage
  Higher voltage can promote faster reduction rate, resulting in smaller size.

- Reaction time
  Higher duration of reaction results in larger particles because longer time reaction results allow for more extensive nucleation and growth.

- Electrolyte concentration
  Higher concentration can lead to more rapid reduction resulting in smaller sized particles.

Even though the method was to synthesis copper nanoparticles, due to high oxidation of copper nanoparticle we obtained $Cu/Cu_2O/CuO$ nanocomposite.

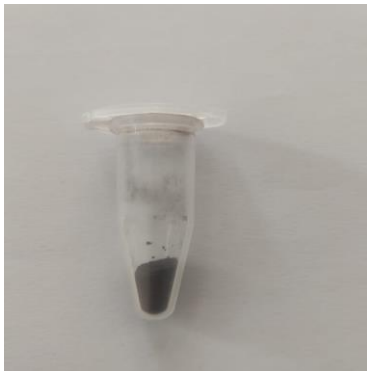

Fig 2.3 Obtained $Cu/Cu_2O/CuO$ nanocomposite.

2.2. Characterization tools

2.2.1. X-ray diffraction

X-ray diffraction is a highly adaptable method for the study of crystalline structure by the analysis of the chemical information. It is used for both quantitative and qualitative analysis of the desired sample. Usually XRD is used for analyzing bulk samples but with developed technology thin films also can to analyzed. The common methods carried out in X-ray diffraction are: Laue's method, powder method and rotating crystal method. This technique follows

the principle of Bragg diffraction.

Bragg diffraction is used to find the interplanar spacing between the crystal planes of a lattice. It is one of the consequences of Laues diffraction. When the sample is irradiated with an X-ray then each atom in the plane of lattices which are parallel form a source for the X-ray to get diffracted. And this diffracted ray interferes with each other to form diffraction pattern from where the required information is collected.

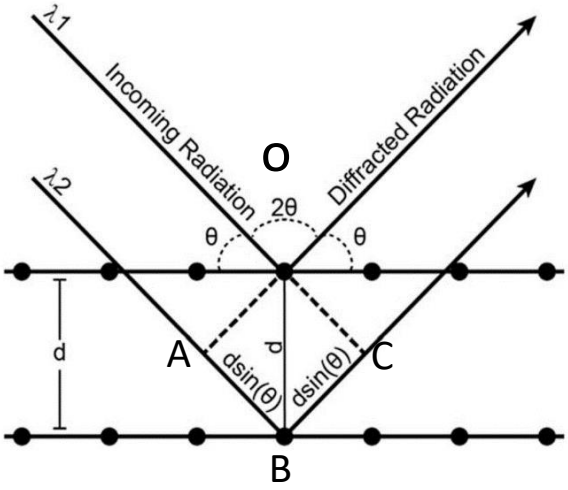

Fig 2.4. Bragg Diffraction

When the X-ray is incident onto a crystal surface, its angle of incidence, θ, will reflect with the same angle of scattering, θ. And, when the path difference, d is equal to multiple of wavelength, λ, constructive interference will occur.

The Bragg equation is given as:

$$n\lambda = 2d \sin\theta \qquad (2.1)$$

The fig shows the waves which are in phase with each other and the diffraction pattern take place when the angle of incidence is equal to angle of diffraction. The incident beam is parallel to each other till they interact with the atom in the plane of lattice. On considering triangle AOB and triangle BOC we find that:

$$AB=BC= d\sin\theta.$$

Then;

$$AB + BC = 2d\sin\theta. \qquad (2.2)$$

And the condition for the diffraction to take place is that the path difference must be an integral multiple of wavelength. And that is given by

$$\Delta = n\lambda \quad (2.3)$$

On equating (2.2) and (2.3) we get the Braggs law as:

$$n\lambda = 2d \sin\theta \quad (2.4)$$

Normally in x-ray diffraction a monochromatic beam of x-rays is used. And coming to the case of monochromator crystal, it consists of a crystal with known lattice spacing and it is oriented in such a way that it only allows the desired wave vectors to pass through it i.e. it acts as a filter. The X-rays are produced in a cathode ray tube and on heating the filament the production of electrons is achieved and these electrons are made to hit the target and the diffraction process take place in two ways either constructively or destructively. Those waves which are in phase interfere to form constructive diffraction. And these diffraction takes place when the Bragg law is satisfied. The geometry of the x-ray spectrometer is in such a way that the target is mounted on a frame at a specific angle θ, and the detector has a flexible arm and it collects the information, this is maintained at an angle 2θ. And intense peaks are observed where there is constructive interference.

2.2.2. UV-Visible absorption spectroscopy

The UV-Visible Spectroscopy deals with the measurement and analysis of the electromagnetic radiations that are absorbed or emitted by atoms or ions of the desired sample is obtained when it moves from ground level to an excited level. The ultra violet region is from 200-400 nm whereas the visible region is from 400-800nm. The instrument associated with this is the spectrophotometer. The spectrophotometer consists of a monochromator, a sample cell, a detector, an amplifier and a recorder. The monochromator is a device that allows and disperses only a certain frequency of desired radiation coming from the source. The radiations which come out through the monochromator are made to impinge the sample. This excites the particles in sample from the ground state to an excited state. In order to become stable and come down to a lower ground state there will a radiative relaxation which causes emission. The sample is kept in a cuvette, made of silica or quartz. Then coming to the detector, photodetectors are used to detect the emitted rays, they are photosensitive. And these rays are analyzed using a recorder.

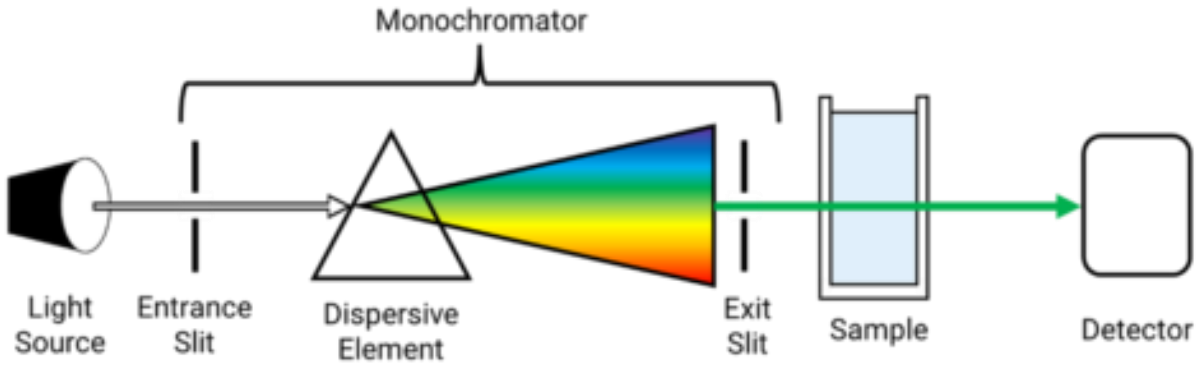

Fig 2.5 Block diagram of UV – Visible Spectrophotometer

2.2.3. Photoluminescence spectroscopy

Photoluminescence method is a non-contact method to investigate the photoluminescent properties of the desired sample. This is a process that is exhibited when light is made to fall on the sample, the phenomenon called photoexcitation takes place. That is when light hits the sample it goes to a higher excited state by gaining certain amount of energy and as it is unstable at the higher energy levels, it comes down to ground state emitting radiative energy. The instrument used to observe this phenomenon is an optical spectrometer. And the emission is analyzed as a function of wavelength. The photoluminescent intensity provides the information about various regions of the material where the composition exhibits excitation energies. The wavelength at which the material has the maximum absorption would be the focus of the photoluminescent spectroscopy. This wavelength will be used to excite the sample to show its emission property. This PL can be used to study numerous properties like finding impurities in the material, to probe the band gap of the material, recombination mechanism etc. The advantage of this characterization is that the sample can be used is any form either solid, liquid or gas. And the wavelength is used in the UV and Visible region (200-800) nm. Some of the factors that are used to describe the properties of PL emission characteristics are the emission wavelength, emission peak bandwidth, intensity and the emission stability. The figure below shows the schematic diagram of working of the photoluminescence spectrometer.

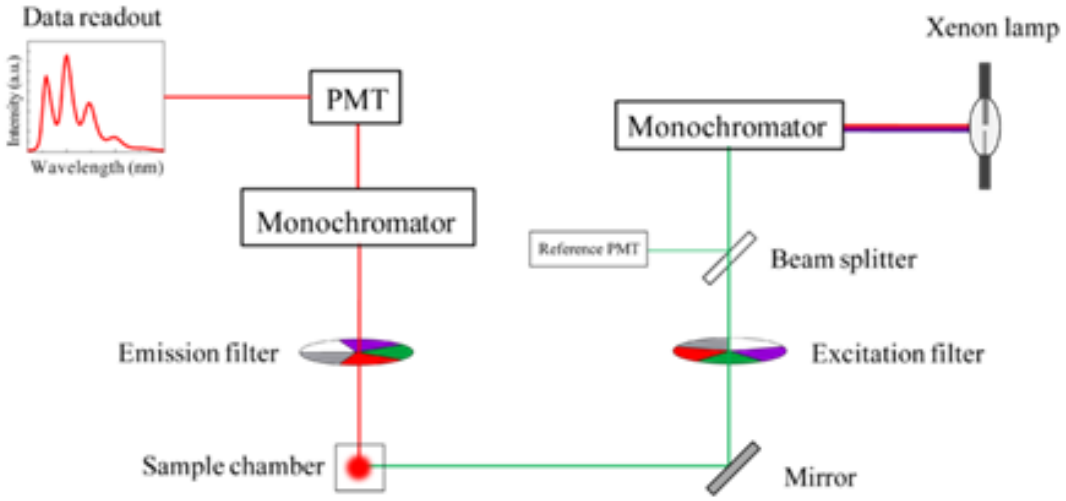

Fig 2.6 Block diagram of Photoluminescent Spectrophotometer

2.2.4. Scanning electron microscopy (SEM)

Scanning electron microscopy is the one of the widely used techniques to find and analyze the nano- and microparticle characterization of solid objects. It helps to analyze the morphological structure of the desired sample. SEM gives the surface shape and composition. The instrument used to carry out this characterization is the scanning electron microscope. Its working is in such a way that electrons of high energies are made to interact with the sample, which in turn gives out a number of signals as a result of the electron and sample interaction. The SEM provides a three-dimensional image of the sample surface. The apparatus consists of an electron gun which acts as the main source and the information that is collected is from the secondary and the back scattered electrons. The samples that are kept inside the vaccum chamber are solids which are able to withstand the given vaccum pressure maintained inside the chamber. The SEM images are highly efficient is producing images that have high resolution and it has a high depth of field. The high magnification is due to the lower wavelengths of the electron beam. The working of this is in such a way that the elastic and inelastic scattering of the incident electrons from the electron gun induces the electrons from the sample to get ejected from the sample surface. Backscattered electrons are ejected as a result of elastic collision with the incident electrons from the electron gun and will

possess energies same as that of incident electrons. Whereas secondary electrons are the ones that are the result of inelastic collision and these electrons will have energies less than the incident electron. The incident electron beam will form a rectangular pattern of parallel lines throughout the sample to produce the desired SEM image. The electron detector collects the ejected electrons from the scanned region. And the resulting SEM image is produced which is in black and white and the brightness on the screen gives the strength of the produced electron signal.

2.3 Thermal lens technique

2.3.1. Introduction

There are many methods to determine thermal diffusivity. One of the traditional methods to measure thermal diffusivity was hot wire technique. But this method had lots of disadvantages. Because of that this method do not produce accurate information. Later many other methods were introduced to measure thermal diffusivity of materials. One such method is thermal lens technique. This method was developed to overcome disadvantages of conventional methods. Thermal lens technique is extremely sensitive because we can measure absorbed energy directly from this technique.

In this technique sample is illuminated by a laser beam having symmetrical intensity distribution. The species in the sample can get excited. When they undergo non-radiative relaxation, they produce heat. Heat is maximum at the center of beam. And a lens-like element will be formed within the sample because of the temperature gradient between center of beam and bulk material. The first thermal lens effect measurement was done by Gordon in 1965. He did this using a single beam apparatus. From there new theories have been derived in order to use thermal lens effect to measure various factors and quantities. Compared to conventional techniques, it has lots of advantages. They have lower background noise, high sensitivity, and has wider applications. In addition to these features, they also has many other features which other techniques do not exhibit. Thermal lens signal depends on the many factors such as - heat that is irradiated within the sample, power of the excitation beam, absorbance of the sample, fluorescence effect of absorbing species. The thermal lens effect will decrease with the increase

in the fluorescence effect. They are also dependent on the thermo-physical properties of sample.

2.3.2. Theory

The non-radiative relaxation of excited species in a sample when illuminated by a laser beam having Gaussian beam profile is called as the thermal lens effect. Non-radiative relaxation includes processes like intersystem crossing, vibrational relaxation and external conversion. When the sample undergoes a non-radiative relaxation, the energy absorbed by the sample will be given out in the form of heat. The temperature of the sample increases because of this. And will reach maximum at the center of beam as the laser has a gaussian beam profile. As a result, there will be change in the refractive index of the sample. The temperature of the sample will decrease with increase in the distance from the center of the beam. And thus, a divergent lens like element will be formed within the sample. Heating of a sample depends on its absorption capability and also the power of the incident laser beam.

2.3.3. Instrumentations

According to the principle of operation, thermal lens technique can be classified in to two – single beam thermal lens technique and dual beam thermal lens technique.

2.3.3.a. Single beam thermal lens technique

In single beam thermal lens techniques, same laser beam is used excite the sample as well as probe the thermal lens effect simultaneously. Such an instrument was initially used in order to determine the dependence of the thermal lens effect on parameters such as laser power, beam divergence, concentration, etc. Though dual beam provides data with better accuracy, single beam technique is still being used for its simplicity in construction and requirement of a single laser. In single beam technique, the laser beam is focused on to the sample through the help of a lens. Shutters or modulators are used for modulating the incoming laser beam. The beam emerging out of the sample, are detected with the help of photodetector which is

placed at a far field. The signal which is detected is amplified and fed to oscilloscope. Any laser beam that is operating in continuous wave mode can be used in single beam technique. The laser should be chosen in such a way that the wavelength of laser beam should be in close approximation with absorption band of the sample. Some of the disadvantages of single beam technique are less accurate data, presence of noise, time consuming, etc.

2.3.3.b. Dual beam thermal lens technique

In dual beam thermal lens technique, two laser beams are used. One of them is known as the pump and other is the probe beam. Pump beam excites the particles in the sample and the probe beam is used to probe the generated thermal lens effect. High power CW or pulsed lasers are normally used as pump lasers on the other hand low powered CW or pulsed lasers are used as probe lasers. In this case two separate lenses are used to focus the beams on to the sample. When pump beam strikes the sample, a divergent lens-like element is formed within the sample. Hence, when probe beam passes through the sample, its intensity is affected. This beam is then fed into the photodetector which is kept at a far field.

2.3.4. Experimental setup

Thermal diffusivity measurements of $Cu/Cu_2O/CuO$ nanofluid were carried out using dual beam thermal lens experiment. Experimental setup requires 2 lasers. One is called as pump and the other one is probe. In our study, DPSS CW laser 488nm and DPSS CW laser 635nm was used as pump and probe.

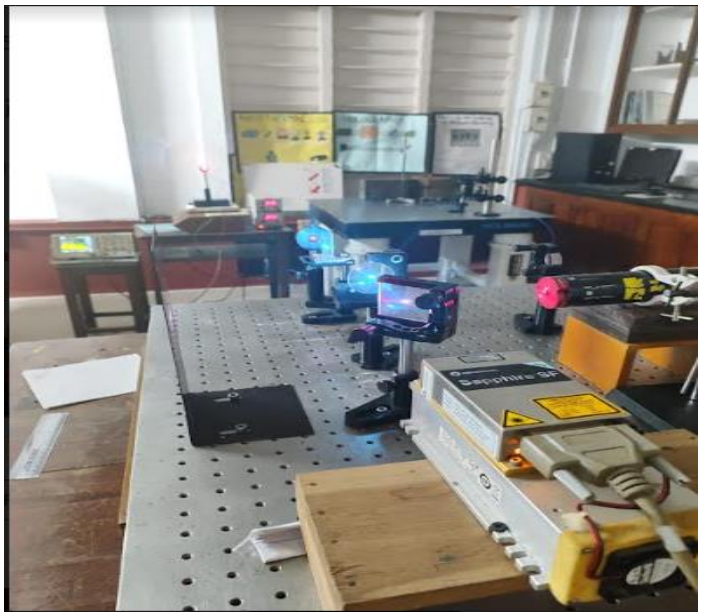

Fig 2.7. Experimental setup

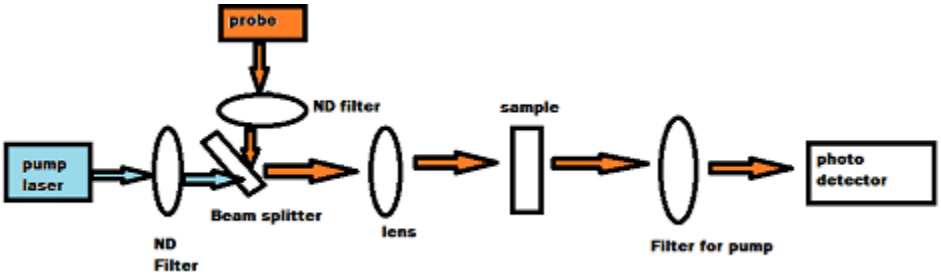

Fig 2.8. Block diagram of dual beam thermal lens technique

A mechanical shutter is used to regulate the intensity of probe laser. Beams that comes from the pump and probe are focused onto the sample which is kept in a cuvette. Pump beam excites the sample. As a result, the sample will undergo a non-radiative relaxation, which releases its energy in the form of heat and the heating effect is maximum at the center of beam. As mentioned above a divergent lens is formed within the sample. The lens formed is called thermal lens. When the probe beam passes through this lens, there will be a change in the intensity of probe beam. This change is detected with the help of a photodetector. The data which is obtained is then mathematically fitted using the equation:

$$I(t) = I_0\left[1 - \frac{\theta}{1 + \frac{t_c}{2(t - t_0)}} + \frac{\theta^2}{2\left(1 + \frac{t_c}{2(t - t_0)}\right)^2}\right]^{-1} \quad (2.4)$$

With the help of MATLAB program t_c and θ was obtained.

θ is a parameter that is associated with the thermal power radiated as heat.

t_c is the characteristic time constant for which focal length of the divergent lens remain constant.

$$t_c = \frac{\omega^2}{4D} \quad (2.5)$$

ω is the size of the beam after passing through the sample. From this equation diffusivity D can be determined.

Initially the setup was standardized with water. Thermal diffusivity D of $Cu/Cu_2O/CuO$ nanofluid at different concentration was found out using the equation

$$D_{sample} = (D_{water} tc_{water})/tc_{sample} \quad (2.6)$$

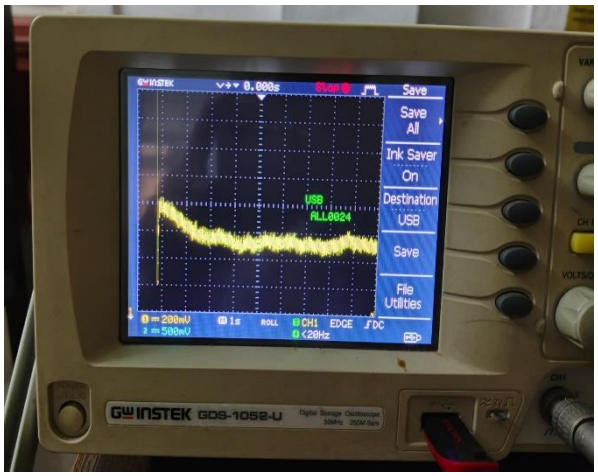

Fig 2.9. Photodetector showing variation in intensity of probe beam.

2.4. Antibacterial study

2.4.1. Introduction

Antibacterial compounds are the agents that prevent the growth of bacteria or kill it without the usage of any toxic substances that affect the surrounding tissue in which the bacteria are present. In order to decrease the growth of bacteria in certain genes, nanoparticles are widely used in present days. The nanoparticles have good pharmaceutical properties. One among them is the use of them in drug delivery to the direct target area. This is a very crucial property as it can reduce any additional side effects and increase the action of the drug at the target region. They are capable for interacting with the surface composed epithelial cells as well. The response of the body to the drugs introduced inside it can be modified by these particles which are in nanometer range. As the frequent use of these nanoparticles emerged, the interest of testing its antibacterial property also increased. And conclusions were made that metallic nanoparticles show appreciable antibacterial properties because of its increased surface area and the presence of a number of reactive sites on the surface.

2.4.2. Copper compounds as antibacterial agents

Copper is a Group 11 element in the periodic table that has an atomic number of 29 and hence there are 29 electrons in one copper atom with a configuration $[Ar]4s^2$ $3d^{10}$. This atomic structure of the copper gives rise to its antibacterial activity. The electrons in the outer most shells play an important role in the activity by acting as donors and acceptors by changing their oxidation state during the reaction. It is the electrochemical potential of the copper ions that results in the suppression activity of copper. The antibacterial activity of copper can happen both inside the target cells and the intercellular spaces. And these were studied using different methods.

2.4.3. The bacteria used for the study

The bacteria used for this study were selected to be E .coli which is gram negative and S. aureus which is gram positive. This causes skin infections in humans and animals. The antibacterial property of copper in these bacteria was investigated.

2.4.4. Antibacterial study of the prepared copper compounds

The antibacterial property of the prepared sample was investigated using the well diffusion method. There are three main steps carried out in this method:

2.4.4.a. Preparation of nutrient media

The nutrient media was prepared by dissolving 1.3g of nutrient broth in 100ml distilled water. 5ml of the nutrient broth taken in test tubes were sterilized using an autoclave. The preparation of nutrient agar media was done by mixing 1.3g of nutrient broth and 2g of agar-agar in 100ml distilled water. This media was then autoclaved and 20 ml of each was poured into sterile petri dishes under aseptic conditions.

2.4.4.b. Preparation of microbial cultures

The test organisms were inoculated into the 5ml of sterilized nutrient broth was kept for incubation at 37^{o} C overnight.

2.4.4.c. Well diffusion method

A lawn culture of each bacterium was prepared using sterilized cotton swabs. A sterilized swab was dipped into the bacterial suspension and moved side to side from top to bottom making sure that all space was covered. The plate was rotated to 90^{o} and the same procedure was repeated so that the entire plate was coated with bacteria. Once the lawn had been prepared, wells of 6 mm diameter were cut into agar plates using a sterile well cutter. The wells were labeled and 20µL of copper

nanoparticles were loaded into corresponding wells. The antibacterial activity of the sample was compared with standard antibiotics available which was Neomycin N30 mcg that is used for antibacterial susceptibility testing. This plate was incubated at 37°C for 24 hrs.

The observations were made by measuring radius of each zone using a standard ruler in millimeters. If the compound is effective against bacteria at a certain concentration, no colonies will grow. This is the zone of inhibition which is a measure of the compound effectiveness, the larger the clear area around the well, the more effective the compound.

CHAPTER 3
RESULT AND DISCUSSIONS

3.1 UV Visible Absorption Studies

The UV-Visible absorption of the synthesized sample was taken and the following was observed. The instrument used for this analysis is spectrophotometer. The result showed peaks at 268nm, 351nm, and 472nm respectively.

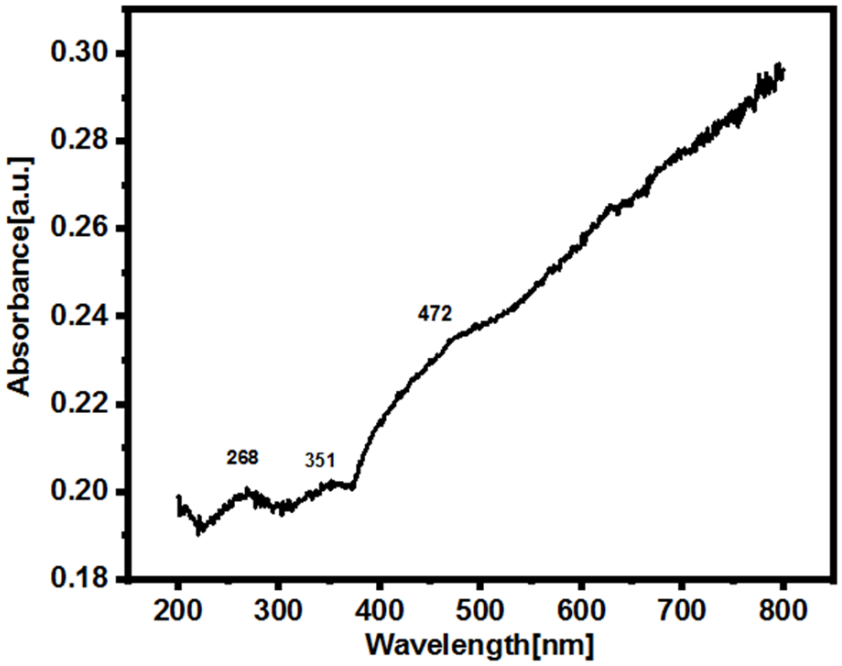

Fig 3.1 Absorption Spectrum of the Cu/Cu_2O/CuO nanocomposite

The peak that is observed at 268nm can be attributed to the presence of CuO in the nanocomposite. In the case of copper nanoparticles, its absorption peak is usually observed at 560nm. But this can vary according to the size of nanoparticles. The peak that is observed at 351nm can be attributed to the presence of small sized copper nanoparticles.
And the peak at 472nm confirms the presence of Cu_2O nanoparticle.

3.2 Photoluminescence Studies

The photoluminescence emission spectrum of the obtained copper mixture was taken by exciting the sample at different excitation wavelengths. The instrument used for this study is spectrophotoflurometer. Fig (a) depicts the emission spectrum of the sample when it was excited at 472nm. The emission peaks for this case was observed at 511nm, 560nm and 707nm. The peak at 707nm indicates the photoluminescence property of Cu_2O nanoparticle. And the other 2 peaks are due to the presence of copper and CuO nanoparticles. Fig (b) shows the emission spectrum of the sample when it is excited at 350nm. The emission peak is seen at 524nm which corresponds to the photoluminescence property of Copper present in the sample and the peak was observed due to the radiative recombination of electron hole pair between the sp-conduction and d-band followed by initial electronic relaxation. Fig (c) depicts the emission spectrum of the sample on exciting it at 260nm wavelength and emission is observed at 398nm which is depicting the photoluminescence property of CuO in the obtained sample, the observed peak at 398nm could be due to the band edge emission.

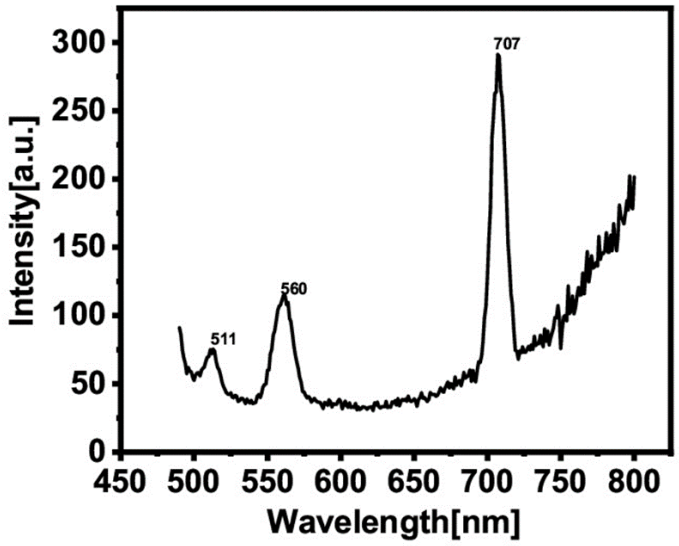

Fig (a)

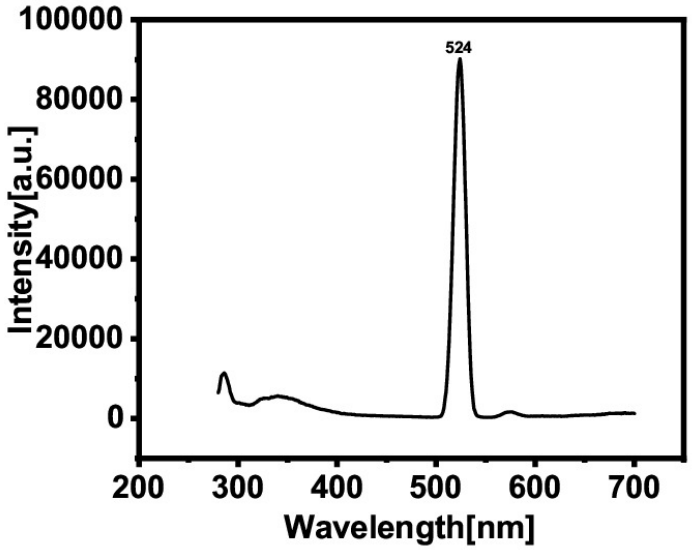

Fig (b)

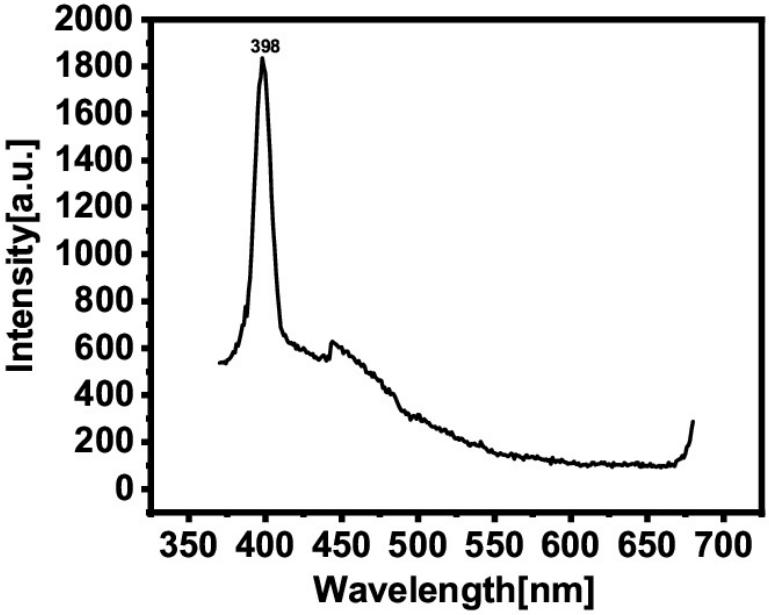

Fig (c)

Fig 3.2. PL spectra of the sample when excited at 472nm, 350nm and 260nm

3.3 Scanning Electron Microscopy (SEM) Studies

Scanning electron microscopy (SEM) is a technique used to get high resolution images by using beams of electrons. When the electron beams interact with the particles in the sample it produces some signals. These signals come and hit the detector to give back and white images. Scanning electron microscope is the instrument used for the SEM analysis.

The structure of the synthesized copper nanoparticle compounds using the electrolysis method was investigated using the SEM analysis.

The figure below shows the SEM image of the sample.

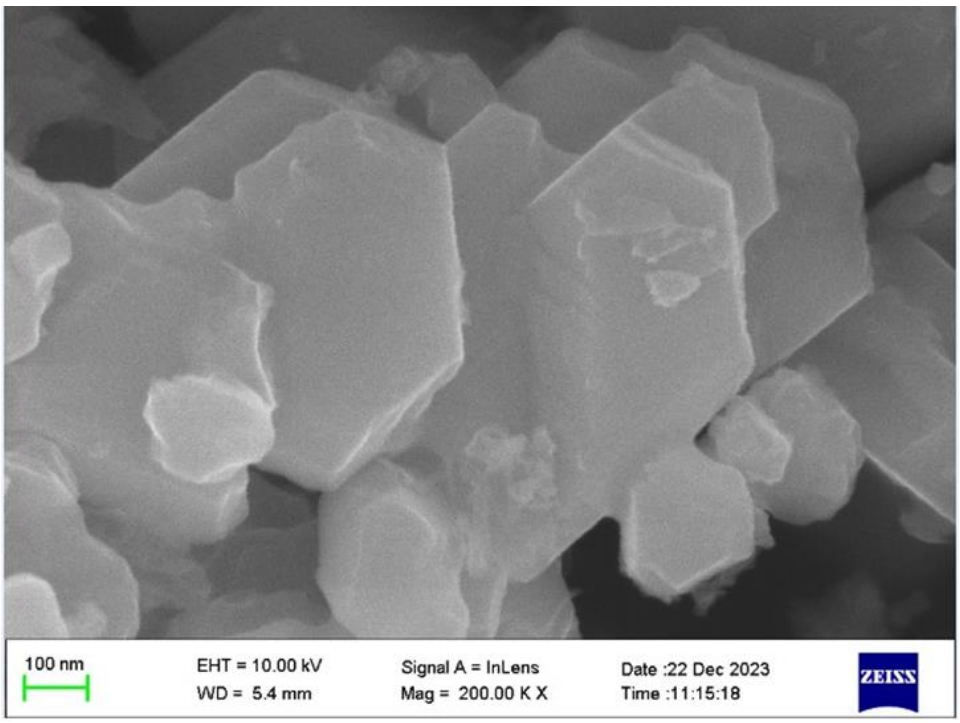

Fig 3.3 SEM image of the sample

The SEM image depicts hexagonal structure of the synthesized copper compound nanoparticles. There are monoclinic and highly crystalline nanoparticles. There are some larger particles to be seen that are formed from small clusters of nanoparticles. The smaller nanoparticles are of the size range below 100nm and those in larger clusters have diameter more than 100nm.The larger composites are

seen in the lower resolution range. In general, metallic crystals as that of Copper and its composites have the tendency to agglomerate due to high tension and surface of the particles in nanoscale range.

3.4 X-Ray Diffraction Studies

The following result was obtained in the XRD for analyzing the properties of the prepared sample.

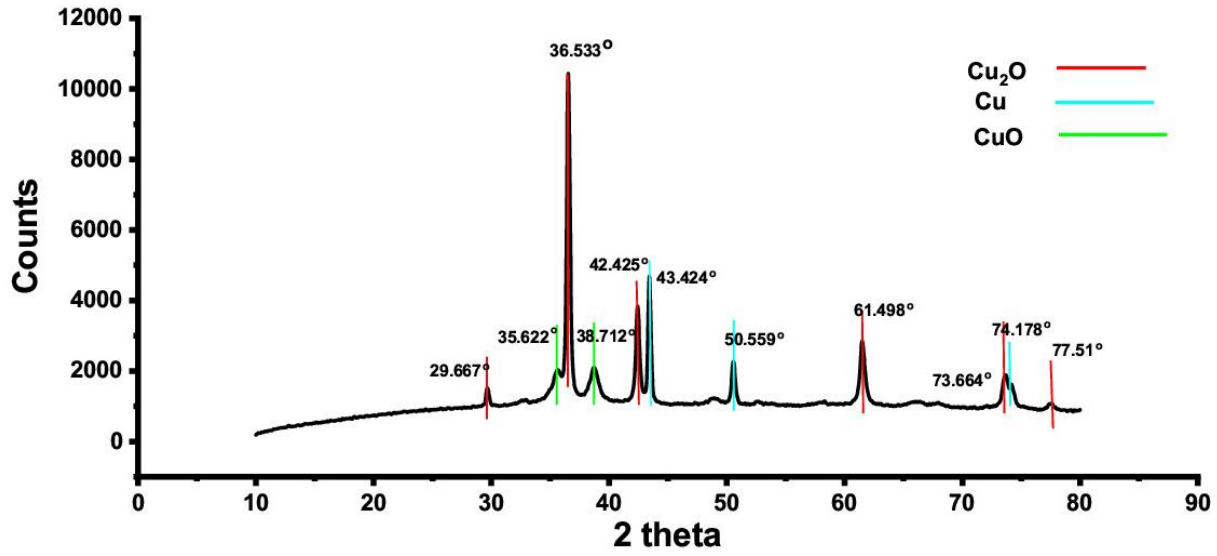

Fig 3.4. XRD of Cu/Cu2O/CuO nanocomposites

It was observed that there is a mixture of Cu, CuO and Cu_2O nanoparticles. And the intense peak corresponds to Cu_2O. The table below shows the peak values i.e. the 2θ values of the desired elements present are:

The peaks that corresponds to Cu_2O are observed at;

29.667, 36.533, 42.425, 61.498, 73.664, 77.51 degrees

The peaks that corresponds to Cu are observed at:

43.424, 50.559, 74.178 degrees

The peaks that corresponds to CuO are observed at:

35.622, 38.712 degrees

The full width half maximum, crystalline size and the corresponding interplanar spacing of Cu, CuO and Cu_2 O nanoparticles present in the nanocomposite are given below:

Table 3.1 crystallite size and interplanar spacing of copper nanocomposites.

Peak Position(2θ) (degrees)	FWHM (β) (radians)	Crystalline Size (D) (nm)	Interplanar Spacing (d) (nm)
43.424	0.28431	31.4016	0.208
50.559	0.3962	26.2389	0.108
74.178	0.57802	17.9888	0.127

Mean (Crystallite Size): 25.290nm

Table 3.2 crystallite size and interplanar spacing of Cu_2O nanoparticles.

Peak Position(2θ) (degrees)	FWHM (β) (radians)	Crystalline Size (D) (nm)	Interplanar Spacing (d) (nm)
29.667	0.29548	29.0383	0.300
36.533	0.32819	26.6183	0.245
42.425	0.33558	26.5167	0.212
61.498	0.49843	19.3632	0.150
73.664	0.58323	17.7679	0.128
77.510	0.62615	16.9991	0.123

Mean (Crystallite Size): 22.71nm

Table 3.3 crystallite size and interplanar spacing of CuO nanoparticles.

Peak Position(2θ) (degrees)	FWHM (β) (radians)	Crystalline Size (D) (nm)	Interplanar Spacing (d) (nm)
35.622	0.68785	12.6653	0.251
38.712	0.54622	16.0949	0.232

Mean (Crystallite Size): 14.380nm

Lattice parameters of copper and Cu_2O nanoparticles are shown below:

Table 3.4 lattice parameters of Cu and Cu_2O nanoparticles

Sample	Interplanar Spacing (d) (nm)	Lattice Parameter Calculated (Å)	Lattice Parameter Standard
Cu (cubic)	1.716	3.598	3.61300
Cu_2O (cubic)	1.93	4.245	4.2685

.

3.5 Photothermal studies

The thermal diffusivity of $Cu/Cu_2O/CuO$ nanocomposite calculated at different concentration is shown below

Table 3.5. Calculated thermal diffusivity for different concentrations

Concentration (µL)	Theta (θ)	t_c (ms)	Diffusivity (D) (x 10^{-8} m^2 s^{-1})
10	-3.0136	208.25	7.395
20	-2.9434	220.478	6.985
30	-2.9077	227.114	6.781
40	-2.835	241.553	6.376
50	-2.8987	228.831	6.730
60	-2.7414	257.742	5.975
70	-2.7414	262.093	5.876
80	-2.8442	239.658	6.426
90	-2.7699	255.614	6.025
100	-2.7886	251.450	6.125

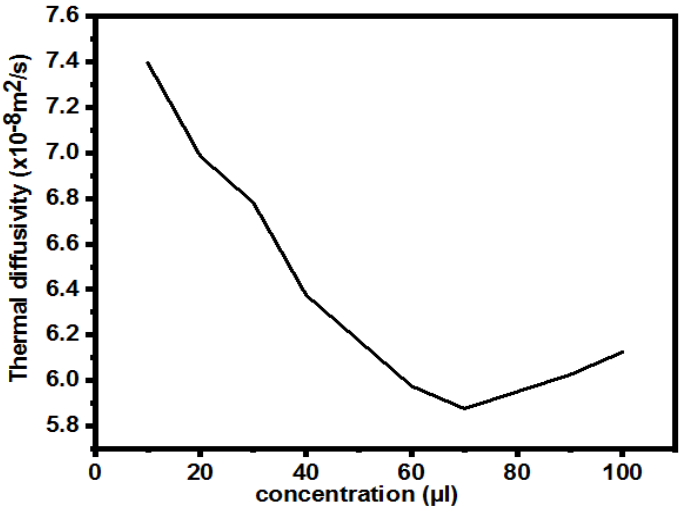

Fig 3.5 Thermal diffusivity of $Cu/Cu_2O/CuO$ nanofluid.

Figure 3.5 shows the result we obtained when calculated thermal diffusivity was plotted against different concentration of $Cu/Cu_2O/CuO$ nanofluid. From the plot it can be seen that initially thermal diffusivity decreases with increase in concentration until it reaches a minimum value, and then increases with increase in concentration.

Initially, as concentration increases, number of particles per unit volume increases. This results in increasing interaction between the particles and thereby reducing the heat exchange between particles and surrounding medium. This is one of the reason for decrease in thermal diffusivity with increase in concentration initially. There is another reason for this as the nanofluid concentration increases there might be tendency for the nanoparticles within the nanofluid to agglomerate or cluster together. This may result in decrease in surface area available for heat transfer and thereby decreasing thermal diffusivity.

But as the concentration continuously increase, the nanoparticles may form an interconnected network within the nanofluid. This network can act as an effective pathway for heat flow, thereby result in increase in thermal diffusivity.

Thermal diffusivity of copper nanoparticles determined using dual beam thermal lens technique in the reviewed literature was found to be $1.2305 \times 10^{-7} m^2/s$ (John J. Mathew, R.J., Rejeena and Mujeeb 2019). But result of this work shows that thermal diffusivity is decreased for $Cu/Cu_2O/CuO$ nanocomposite and the maximum value is found to be $0.7395 \times 10^{-7} m^2/s$.

3.6 Antibacterial study

The inhibition of the prepared compound in each bacterium is shown in the figures below. There are two types of bacteria used gram positive and gram negative. The gram-positive bacteria (S. aureus) does not have an outer membrane instead it is surrounded by many thicker layers of peptidoglycan. The gram-negative bacteria (E. coli) has a thin wall of peptidoglycan which is further covered by an outer membrane that contains lipopolysaccharide.

Fig 3.6 antibacterial activity against E.coli.

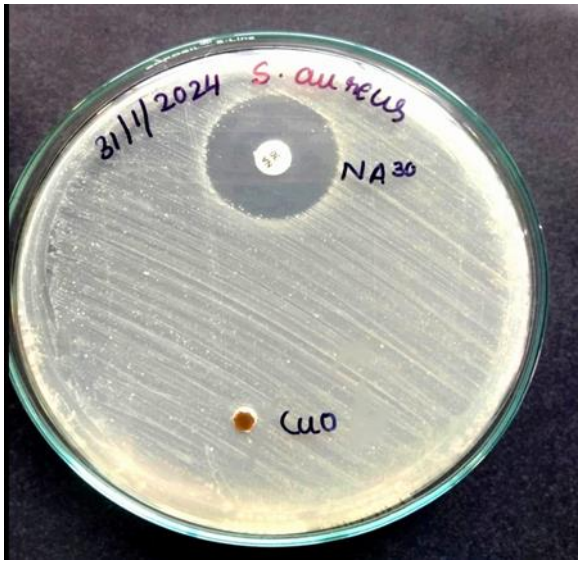

Fig 3.7 Antibacterial activity against S. aureus

Table 3.6 Zone Of Inhibition of the nanocomposite samples against E. coli and S. aureus

Microorganism	Zone of Inhibition(mm)
E. coli	10
S. aureus	Nil

•The prepared copper compound nanoparticles have been found to exhibit antibacterial activity in E-coli bacterium. The inhibition zone of E-coli was 10mm.

•On the other hand, there was no activity of Cu/CuO/Cu_2O nanocomposite in S. aureus

The inhibition zone in the literature reviewed has 15mm for E. coli and 8-10mm for S. aureus. (Theivasanthi and Alagar 2011). For understanding the antimicrobial activity of copper composite on the respective gram-positive and gram-negative bacteria , it is necessary to the know how these bacteria grow. The copper composite particle will make the enzyme inactive by stopping its interaction with oxygen and thus it suffocates the bacteria and the cell is destroyed without causing any damage to the surrounding tissues present. The structure of gram-negative bacteria is in such a way that it does not have an outer membrane and because of this reason the synthesized copper compounds can easily fuse and penetrate into the bacterial cell and the copper ions start to form a thick layer around the cell stopping the cytoplasm to grow further. In the reviewed paper it is seen that E-coli due to its structure is easily disrupted by the copper ions which is introduced in it. (Mohammed Rafi and Saba Mehrwan 2010). And in the case of S. aureus no inhibition zone is due to the thick peptidoglycan layer. And thus, it is evident that the formation of copper ion barrier is important to suppress the growth of bacteria.

CHAPTER 4
CONCLUSION

In this work, $Cu/Cu_2O/CuO$ nanocomposites were synthesized through electrolysis method. It was then characterized structurally, morphologically and optically.

In UV-Visible spectrum, three peaks at 268nm, 351nm and 472nm was observed. Each peak correspond to different nanoparticles present in the nanocomposite. Peak at 268nm corresponds to the presence of CuO nanoparticles. And the other 2 peaks attribute to the presence of Cu and Cu_2O nanoparticles.

In the case of photoluminescence spectroscopy, the sample was excited at 260nm, 350nm and 472nm. When excited at 260nm, emission peak at 398nm was observed. Emission peak was observed at 524nm when the sample was excited at 350nm and 3 peaks at 511nm, 560nm, and 707nm were observed when the sample was excited 472nm.

From the XRD pattern of $Cu/Cu_2O/CuO$ nanocomposite, the crystallite size, interplanar spacing and lattice constants of each element present in the nanocomposite was calculated and found to be in good agreement with the observed data. The average particle size of each nanoparticle was found to be in nano range.

Hexagonal structures were observed in SEM analysis.

The photothermal as well as antibacterial study of the sample was done. In photothermal study we found that thermal diffusivity of the $Cu/Cu_2O/CuO$ nanocomposite initially decreases with increasing concentration and after reaching a minimum value then it increases with increasing concentration. Antibacterial study was carried out in E. coli and S. aureus. An inhibition zone of 10mm in the case of E.coli was observed. On the other hand, no inhibition zone was observed in the case of S. aureus.

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