

PROJECT REPORT

On

GREEN SYNTHESIS OF COPPER OXIDE NANOPARTICLES: CHARACTERIZATION AND APPLICATIONS

Submitted by

**ALISHA SUSAN JOSHY
(AB21CHE012)**

In partial fulfilment for the award of the

Bachelor's Degree in Chemistry



DEPARTMENT OF CHEMISTRY AND CENTRE FOR RESEARCH

**ST. TERESA'S COLLEGE (AUTONOMOUS)
ERNAKULAM**

2023-2024

DEPARTMENT OF CHEMISTRY AND CENTRE FOR RESEARCH

ST. TERESA'S COLLEGE (AUTONOMOUS)

ERNAKULAM



B.Sc. CHEMISTRY PROJECT REPORT

Name : ALISHA SUSAN JOSHY
Register Number : AB21CHE012
Year of Work : 2023-2024

This is to certify that the project "**GREEN SYNTHESIS OF COPPER
OXIDE NANOPARTICLES: CHARACTERIZATION AND
APPLICATIONS**" is the work done by ALISHA SUSAN JOSHY.

Saritha
Dr. Saritha Chandran A.
Head of the Department



Linto Anto
Mr. Linto Anto
Staff-member in charge

Submitted to the Examination of Bachelor's Degree in Chemistry

Date: *4/5/24*

Examiners:

Dr. Ajith James Jose SB College Changanassery
Dr. Nisha T.P. *JR-2024* *Jull*

DEPARTMENT OF CHEMISTRY AND CENTRE FOR RESEARCH

**ST. TERESA'S COLLEGE (AUTONOMOUS)
ERNAKULAM**



CERTIFICATE

This is to certify that the project work entitled “**GREEN SYNTHESIS OF COPPER OXIDE NANOPARTICLES: CHARACTERIZATION AND APPLICATIONS**” is the work done by **ALISHA SUSAN JOSHY** under my guidance in the partial fulfilment of the award of the Degree of Bachelor of Science in Chemistry at St. Teresa's College (Autonomous), Ernakulam affiliated to Mahatma Gandhi University, Kottayam.

Mr. LINTO ANTO

Project Guide

DECLARATION

I hereby declare that the project work entitled “**GREEN SYNTHESIS OF COPPER OXIDE NANOPARTICLES:CHARACTERIZATION AND APPLICATIONS**” submitted to Department of Chemistry and Centre for Research, St. Teresa’s College (Autonomous) affiliated to Mahatma Gandhi University, Kottayam, is a record of an original work done by me under the guidance of **Mr. LINTO ANTO, ASSISTANT PROFESSOR,** Department of Chemistry and Centre for Research, St. Teresa’s College (Autonomous), Ernakulam and this project work is submitted in the partial fulfilment of the requirements for the award of the Degree of Bachelor of Science in Chemistry.

ALISHA SUSAN JOSHY

Acknowledgements

The success and final outcome of this project required a lot of guidance and assistance from many people, and we were extremely fortunate to have this all along the course of our project work. First of all, we thank the God Almighty for being with us throughout all the days and helping us to complete the project successfully. We respect and thank our project guide Mr. Linto Anto, Assistant Professor, Department of Chemistry, St. Teresa's College (Autonomous), Ernakulam for introducing us to the world of research and opportunities. We thank her for her guidance and constant supervision and motivation in the successful completion of the project. We extend our sincere gratitude to Dr. Sr. Vinetha, Director, Dr. Alphonsa Vijayan, Principal, Dr. Saritha Chandran A., Head of the Department of Chemistry, and all the faculty members of the Department of Chemistry, St. Teresa's College (Autonomous), Ernakulam for providing us the infrastructure required for this project and their well wishes. We would like to thank all the teachers and non-teaching staffs of the Department of Chemistry,

St. Teresa's College, Ernakulam for their support and co-operation during our entire project. We also feel proud to have a group of good friends who offered us much needed help in completing this project.

ALISHA SUSAN JOSHY

Contents

Chapter 1 Introduction	1
1.1 Nanochemistry	1
1.1.1 Nanoparticles	2
1.1.2. Uses of nanoparticles	2
1.2 Copper oxide	3
1.2.1 Uses	4
1.3 Copper oxide nanoparticles	5
1.3.1 Advantages of copper oxide nanoparticles over copper oxide	5
1.3.2 Synthesis of copper oxide nanoparticles	5
1.3.3 Characterization of copper oxide nanoparticles	8
1.3.4 Application of copper oxide nanoparticles	11
1.4 Scope and objectives of the present work	13

Chapter 2 Materials and Methods	14
2.1 Materials	14
2.1.1 Copper sulphate pentahydrate	14
2.1.2 Sodium hydroxide pellets	14
2.1.3 Eriochrome Black T	14
2.2 Experimental methods	14

2.2.1	Preparation of leaf extract	14
2.2.2	Synthesis of copper oxide nanoparticles	15
2.2.3	Characterization	16
2.2.3.1	Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX)	16
2.2.3.2	Infra-red spectroscopy (IR)	16
2.2.3.3	X-ray diffraction	16
2.2.4	Application	17
2.2.4.1	Dye adsorption of Eriochrome Black T	17
Chapter 3 Results and Discussion		18
3.1	Characterization of copper oxide nanoparticles	18
3.1.1	Infra-red spectroscopy	18
3.1.2	SEM-EDX	19
3.1.3	XRD	21
3.2	Application	22
3.2.1	Adsorption of Eriochrome Black T	22
Chapter 4 Conclusions		24
References		25

Chapter 1

Introduction

1.1 NANOCHEMISTRY

Nano-chemistry is a subfield of nanoscience concerned with the chemical applications of nanomaterials in nanotechnology. Nano-chemistry is a relatively new branch of chemistry that focuses on the study and development of methods for preparing useful materials with nano-meter-sized dimensions (1-100nm). Nano-chemistry is a branch of solid-state chemistry that emphasizes the synthesis of building blocks based on size, surface, shape, and defect properties rather than the production of matter itself. Atoms can be manipulated physically and chemically to form molecules and nanoscale assemblies. Nano-chemistry is the study of the synthesis and characterization of nanoscale materials.

Nano-chemistry is the science of tools, technologies, and methodologies for novel chemical synthesis, such as using synthetic chemistry to create nanoscale building blocks with desired (prescribed) shape, size, composition, and surface structure, as well as the potential to control the actual self-assembly of these building blocks to various desirable sizes. Because of their small size, nanoparticles have 'unusual' structural and optical properties that can be used in catalysis, electro-optical devices, and other applications.

Nanomaterials have structural properties that are intermediate between atoms and bulk materials. The properties of nanometre-scale materials differ significantly from those of atoms and bulk materials. Nanomaterials have an extremely large surface area to volume ratio due to their small

dimensions, resulting in more 'surface dependent' material properties. Because the surface properties of nanoparticles are the most important criteria to consider for applications.

1.1.1. Nanoparticles

Nanoparticles are particles of matter with diameters ranging from 1 to 100 nanometres (nm). Chemical reactivity, energy absorption, and biological mobility are some of the properties that distinguish nanoparticles from bulk materials simply by virtue of their size. Nanoparticles have different physical, chemical, and biological properties than their larger counterparts. Nanoparticles can be classified into several types based on their size, shape, and material properties. Some classifications differentiate between organic and inorganic nanoparticles; the former includes dendrimers, liposomes, and polymeric nanoparticles, while the latter includes fullerenes, quantum dots, and gold nanoparticles. Other classifications divide nanoparticles based on whether they are carbon-based, ceramic, semiconducting, or polymeric. Furthermore, nanoparticles can be hard (e.g., titania [titanium dioxide], silica [silica dioxide] particles, and fullerenes) or soft (e.g., liposomes, vesicles, and nanodroplets). The classification of nanoparticles typically depends on their application, such as in diagnosis or therapy versus basic research, or may be related to how they were produced.

1.1.2 Uses of nanoparticles

- Nanoparticles are used to make scratch-resistant eyeglasses, crack-resistant paints, anti-graffiti coatings for walls, transparent sunscreens, stain-repellent fabrics, self-cleaning windows, and ceramic coatings for solar cells.
- Nanoparticles used as fillers in tyres can improve adhesion to the road, reducing stopping distance in wet conditions. The stiffness of the car body can be increased by using nanoparticle-enhanced scale.

- Nanostructured ceramic coatings are far tougher than traditional wear-resistant coatings for machine parts. Nanotechnology-enabled lubricants and engine oils also significantly reduce wear and tear, extending the lifetimes of moving parts in everything from power tools to industrial machinery.
- Nanoparticles can also help reduce electronics' power consumption, reduce the weight and thickness of electronic screens, and increase computer speed.
- Nanoparticles are used in a wide range of health products and medical treatments. Nanoparticles and nanotechnology are used in products such as sunscreen, deodorant, and cosmetics. They are used in household products such as stain removers, degreasers, and air filters and purifiers, as well as stain-resistant paint for your walls.
- Nanoscale additives applied to fabric surface treatment can provide lightweight ballistic energy deflection in personal body armour.
- Nanomaterials are also used in biology and medicine in a variety of ways, including direct product application into patients. Examples include drug delivery and gene therapy products, biological molecule and cell separation and purification, fluorescent biological labels, imaging contrast agents, tissue engineering, DNA probes and nanoscale biochips, and microsurgical technology.

1.2 Copper oxide

Copper oxide is also known as cuprous oxide, is covalent in nature. The solid is diamagnetic. It is mainly used as antimicrobial agents. The health of humans depends on copper because it is less toxic and essential. Compared to other metals, copper is less expensive and is frequently used to create nanoparticles that boost the potency of antibiotics. Copper nanoparticles have a high area to volume ratio. When compared to other

metals like gold and silver, copper has greater antibacterial activity, catalytic potency, magnetic, and optical properties.

1.2.1 USES

Copper oxide is an efficient corrosion inhibitor and is used in antifouling paints for ship and boat bottoms. It is utilized in porcelain and glass paints. It is used as a p-type semiconductor material to create rectifiers and photocells for light meters. And it is also used as a seed dressing and fungicide. In medical industry, copper oxide has a wide range of uses; copper oxide containing antiviral respiratory masks that lower the risk of infection, wound dressings that promote wound healing, acaricidal bedding items that kill dust mites and athlete's foot-protecting socks have been created. They are also used in high-tech superconductors, semiconductors, and solar-energy conversion. They can be implemented in thermoelectric materials, catalysts, superconducting materials, glass, sensing materials, ceramics, and a variety of other applications.

Biological role:

Copper oxide nanoparticles are widely used as anticancer, antibacterial, and antioxidant agents because of their ability to interact with cellular components and participate in multiple biological reactions and functions. Enzymes that are involved in aerobic metabolism, Lysy oxidase in connective tissue, dopamine monooxygenase in the brain, and cytochrome c oxidase in mitochondria, require copper. Copper ions are necessary for the proper functioning of cells while it is poisonous. Copper ions are necessary for several enzymes and are used in respiration, neuronal transmission, tissue maturation, defence against oxidative stress, and iron metabolism, among other cellular functions.

1.3. COPPER OXIDE NANOPARTICLES

Copper oxide nanoparticles are mainly used as antimicrobial agents. It has a particle size range of 20-95 nm. Copper oxide nanoparticles have stable chemical and physical characteristics, are relatively inexpensive, and are photocatalytic. Copper oxide nanoparticles have the potential to be used as anti-infective agents to their desirable crystal morphologies and incredibly high surface area. Synthesised CuO nanoparticles have brownish-black colour. It is insoluble in water; alcohol or ammonia solution are mainly used as the solvents for CuO nanoparticles.

1.3.1 Advantages of Copper oxide Nanoparticles over copper oxide

Copper Oxide receives huge attention due to its salient features such as structure, band gap and chemical properties. However, the very attractive properties of bulk copper oxide can be significantly improved by the preparation of the CuO nanostructures. They can be used as antimicrobial, antifungal agents, as they inhibit the growth of microorganisms such as bacteria, fungi, viruses, and algae. The size of nanoparticles and the high surface zone of copper oxide permit them to interact closely with the cell membrane and thus have an excellent antimicrobial potential.

1.3.2 Synthesis of Copper oxide Nanoparticles

There have been reported various approaches of synthesizing CuONP's. The different methods include physical, chemical and biological methods. It has also been reported that the method of CuONP's synthesis affect their morphological properties and toxicity behaviour. The most common used techniques in the physical approach of CuONP's synthesis are electro-spraying, laser pyrolysis, laser ablation and evaporation-condensation. Among the physical approach, pulsed laser induced ablation technique had gained more attention owing to the fact that it's easy, eco-friendly and uniformly nanoparticles are obtainable. The advantages of the physical

method are production of CuONP's with uniform, controlled sized and high purity. In chemical method it makes use of some chemicals/regents for the reduction of copper ions during the synthesis of CuONP's. Chemical approach of nanoparticles formation is grouped into two forms; green chemical approach and traditional approach. For the green chemical approach, chemicals synthesized from organic material such as ascorbic acid are used while inorganic compounds such as sodium borohydride and potassium borohydride are used in the traditional approach. The use of ascorbic acid as a reducing agent and the use of potassium borohydride in formation of CuONP's has been established. Many of these reducing agents have been associated with environmental toxicity or biological hazards. Several reports had shown that large energy consumption, environmental pollution, the use of high pressure and temperature, expensive and toxic chemicals as huge limitations of chemical method of synthesizing CuONP's and other transitional metal oxide NPs¹.

With an increasing interest in the minimization or total elimination of waste and the execution of sustainable processes through the implementation of the fundamental principles of green chemistry, the development of natural and biomimetic approaches for the preparation of nanomaterials is a desirable aspect. Hence, there is a need to develop newer ecofriendly processes for the synthesis of metal/metal. So, we opted the leaf of plant *Coleus Amboinicus* for the synthesis of copper oxide nanoparticles. Green synthesis of CuONP's using plant extracts as the source of electron generation for the reduction of copper salt display some advantages over the use of microbes because it does not require cell culture maintenance and it can be scaled up for large-scale synthesis. The formation of CuONP's occurs with an observable change in the colour of the extract when copper salt was added. Several studies have revealed that the phytochemicals in the

plant extracts first form complexes with the ion salts and then reduce the ions to form nanoparticles. The biomolecule in the plants extracts usually react with copper ion to cause reduction which subsequently transform into CuONP's

Here we used copper sulphate pentahydrate as the metal precursor and *Coleus amboinicus* leaf extract as the reducing agent. In this work, we present a green method for the synthesis of copper oxide nanoparticles using plant extract of *Coleus Amboinicus*.



Fig 1.1 coleus amboinicus

Coleus Amboinicus- is now called as *Plectranthus amboinicus*, is a semi succulent perennial plant belonging to the family *Lamiaceae* with a pungent oregano-like flavour and odour. The plant is commonly called as Indian borage or Indian mint. It is native to Southern and Eastern Africa and widely cultivated in the tropics where it is used as a traditional medicine, spice and ornamental plant. Leaves are 5–7 cm, by 4–6 cm, fleshy, undivided (simple), broad, egg/oval-shaped with a tapering tip. The copper oxide nanoparticles synthesized here is used to investigate its dye adsorption property².

1.3.3 Characterization of Copper Oxide Nanoparticles

X-RAY DIFFRACTION

X-ray Diffraction (XRD) is a powerful analytical technique used to study the crystalline structure of materials. It is used in various scientific and industrial fields to determine the arrangement of atoms within a crystalline substance, providing valuable information about the material's crystallography, crystal size, orientation, and phase composition. X-rays are generated using an X-ray tube and directed at the sample, which is in a crystalline form. When X-rays strike the sample, they interact with the crystal lattice, causing constructive interference and scattering in different directions. This results in a diffraction pattern, which is recorded by a detector.

The resulting diffraction pattern contains information about the spacing of crystal planes within the sample, which is converted into a diffraction profile, known as the X-ray diffraction pattern or X-ray diffraction spectrum. Scientists can analyse the XRD pattern to determine the crystal structure of the material, including lattice parameters, unit cell dimensions, and atom arrangement.

XRD is used in various applications, including material characterization, pharmaceuticals, geology, chemistry, materials science, and quality control. It is a non-destructive technique that provides detailed information about the internal structure of materials and is a fundamental tool in various fields.

FOURIER TRANSFORM INFRARED SPECTROSCOPY

Fourier-Transform Infrared Spectroscopy (FTIR) is a widely used analytical technique in chemistry and materials science for identifying and characterizing the chemical composition of substances. It measures the interaction of infrared radiation with matter, which is in the electromagnetic

spectrum just beyond the visible light range. The principle behind FTIR spectroscopy is that molecules vibrate at characteristic frequencies, and when infrared light passes through a sample, some of it is absorbed by the sample, resulting in a plot of the sample's absorbance as a function of the frequency of the infrared light. This absorption is related to molecular vibrations and can provide information about the functional groups and chemical bonds present in the sample.

The sample can be a gas, liquid, or solid. In a conventional FTIR spectrometer, an interferometer is used to modulate the infrared light, and a detector measures the resulting interference pattern. A computer then performs a Fourier transformation on the interferogram to obtain the infrared spectrum, which shows peaks at specific wavelengths associated with the sample's chemical constituents.

FTIR spectroscopy is used in a wide range of applications, including the identification of unknown substances, quality control in manufacturing, environmental analysis, forensic science, pharmaceuticals, and monitoring chemical reactions. It provides qualitative and quantitative information about the chemical composition of a sample.

The infrared region of the electromagnetic spectrum is divided into three main regions: the near-infrared (NIR), mid-infrared (MIR), and far-infrared (FIR). FTIR spectroscopy primarily deals with the mid-infrared range, which is generally between 4000 and 400 cm^{-1} (wavenumbers).

In conclusion, FTIR spectroscopy is a versatile and powerful analytical tool used in various fields of science and industry for material characterization, quality control, and research purposes. It provides valuable information about the chemical structure and composition of a wide range of substances, making it a valuable tool in various fields.

SCANNING ELECTRON MICROSCOPY – EDAX

Scanning Electron Microscopy (SEM) is a highly advanced technique used in various scientific and industrial fields for high-resolution imaging and analysis of the surface morphology and composition of materials. SEM works by scanning a focused beam of high-energy electrons across the sample's surface, generating signals such as secondary electrons (SE) and backscattered electrons (BSE). These signals provide information about the sample's surface, including its topography and texture.

Sample preparation is crucial for SEM analysis, with samples typically coated with a thin layer of conductive material to prevent charging effects and improve image quality. The emitted electrons are detected and used to create high-resolution images of the sample's surface, providing detailed information about the morphology, microstructure, and surface features.

SEM applications include material science, analysing metals, ceramics, polymers, composites, and minerals, and geology, examining minerals and rocks. Energy-Dispersive X-ray Analysis (EDAX), also known as Energy-Dispersive X-ray Spectroscopy (EDS or EDX), is a technique used in electron microscopy and related analytical instruments to determine the elemental composition of a sample by detecting characteristic X-rays emitted when the sample is bombarded with electrons.

EDAX collects characteristic X-rays emitted by the sample and produces an energy spectrum, which contains peaks at specific energies corresponding to the elements present in the sample. The intensity of these X-ray peaks is proportional to the concentration of the corresponding elements in the sample, providing information about the elemental composition of the sample. EDAX is essentially another term for EDS, providing information about the elemental composition of a material in conjunction with SEM.

UV VISIBLE ABSORPTION SPECTROPHOTOMETER

UV-Visible Absorption Spectroscopy is a widely used analytical technique for studying the absorption of ultraviolet and visible light by molecules and atoms in a sample. It provides valuable information about the electronic structure, concentration, and chemical composition of substances. The principle of UV-Vis Spectroscopy is that molecules and atoms can absorb light at specific wavelengths when the energy of the incoming photons matches the energy difference between the ground state and an excited state. The key instrument used for this technique is a UV-Vis spectrophotometer, which consists of a light source, a monochromator, a sample holder or cuvette, a detector, and a computer for data analysis. The sample is typically dissolved in a suitable solvent, and its absorbance is measured in a cuvette. The spectrophotometer measures the intensity of the incident and transmitted light, and the difference in intensity is used to calculate the absorbance (A) of the sample.

The absorbance data is used to construct an absorption spectrum, which is a plot of absorbance (A) against the wavelength (or frequency) of the incident light. UV-Vis Spectroscopy is used for both qualitative and quantitative analysis, allowing for the determination of the concentration of a particular substance in the sample.

Applications of UV-Vis Spectroscopy include quantitative analysis, qualitative analysis, chemical kinetics, biology and biochemistry, environmental analysis, and pharmaceuticals.

1.3.4 Application of Copper Oxide Nanoparticles

Copper oxide (CuO) nanoparticles are small, high-surface area particles made up of copper and oxygen atoms. They have unique properties due to their small size and high surface area, making them useful in various

scientific and industrial applications. CuO nanoparticles typically have a size range of 1 to 100 nanometre's, allowing them to have a large surface area and quantum size effects. They can have different crystal structures, with monoclinic being the most common, which affects their properties and reactivity. Their high surface area-to-volume ratio makes them highly reactive, suitable for catalytic and sensing applications. They also exhibit optical properties, including light absorption in ultraviolet and visible regions.

1. Catalysis³: CuO nanoparticles are used as catalysts in a variety of chemical reactions, including the oxidation of organic compounds, hydrogenation, and reduction reactions.
2. Gas Sensors⁴: Due to their high surface area and sensitivity to certain gases, CuO nanoparticles are used in gas sensors for detecting harmful gases like carbon monoxide (CO) and ammonia (NH₃).
3. Solar Cells⁵: CuO nanoparticles have been investigated for use in solar cells and photovoltaic devices due to their ability to absorb light in the ultraviolet and visible range.
4. Batteries and Supercapacitors⁶: They are explored for energy storage applications, including lithium-ion batteries and supercapacitors, to enhance energy storage and charge/discharge rates.
5. Antimicrobial Coatings⁷: CuO nanoparticles are incorporated into coatings and textiles to provide antimicrobial properties, helping to inhibit the growth of bacteria and fungi.
6. Photocatalysis⁸: CuO nanoparticles can be used in photocatalytic applications, such as water purification and pollutant degradation when exposed to UV or visible light.

7. Nanofluids⁹: CuO nanoparticles are added to fluids, creating nanofluids that have enhanced thermal properties, making them useful in cooling and heat transfer applications.

8. Optoelectronics¹⁰: They are explored for use in optoelectronic devices, including light-emitting diodes (LEDs) and sensors.

1.4 OBJECTIVES

- To synthesis CuONP's using *Coleus Ambonicus* leaf extract and copper sulphate as a precursor.
- Characterization of synthesized nano particles using spectroscopic methods.
- To study the adsorption of dye using synthesized nano particles.

Chapter 2

Materials and Methods

This chapter gives a brief description of the materials and experimental procedures adopted for the present investigation.

2.1 MATERIALS

2.1.1 Copper sulphate pentahydrate manufactured Copper sulphate pentahydrate by Merck Specialities Private LTD. Mumbai.

2.1.2 sodium hydroxide pellets

Sodium hydroxide pellets manufactured by Nice Chemicals (P) LTD. Edappally, Kochi were used for the study.

2.1.3 Eriochrome Black T

Eriochrome black T manufactured by Nice Chemicals (P) LTD. Edappally, Kochi were used for the study.

2.2 EXPERIMENTAL METHODS

2.2.1 Preparation of leaf extract

The leaves of the plant *coleus amboinicus* were collected from the terrace of an apartment located at Ernakulam. The weight of the leaves required for the synthesis was about 20g (5 to 6 medium sized leaves). After weighing the leaves, they were washed thoroughly and crushed using a mortar and pestle. These crushed leaves were then transferred into a 250 ml beaker containing 100ml of distilled water. The above mixture was kept in an oven for 100 °C for 20 minutes. It was then stirred using a magnetic stirrer for

20 minutes. This was cooled to room temperature and filtered using a filter paper. The filtrate was used as the leaf extract¹¹.

2.2.2 Synthesis of copper oxide Nanoparticles

12.48g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ was weighed and taken in a standard flask and made up to 50 ml. Thus 1M of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ was prepared. Next 15ml of the prepared leaf extract was mixed with the above solution and was stirred using a magnetic stirrer for 30 minutes at 100°C . After that it was kept in an oven for 45 minutes at 100°C . After taking it from oven, 1M of NaOH solution was added dropwise while stirring. The NaOH solution was added until brownish black color appeared and was again stirred for another 30 minutes. This was then centrifuged 3 times at 1200 rpm for 2 minutes and filtered using WhatmanNo.1 filter paper. The precipitate obtained was placed in a crucible and this was kept in muffle furnace for 30 minutes at 180°C ¹¹. Hence the copper oxide nanoparticles were synthesized (fig 2.1)



Fig 2.1 Copper oxide nanoparticles

2.2.3 CHARACTERIZATION

2.2.3.1 Scanning Electron Microscopy with Energy Dispersive X-ray spectroscopy (SEM-EDX)

SEM -EDX were taken using Jeol 6390LA/ OXFORD XMX N spectrometer with

Accelerating voltage: 0.5 to 30 Kv, Filament: Tungsten, Magnification x 300000,

EDAX resolution 136 eV, EDAX detector area 30 mm² and Elemental Mapping.

2.2.3.2 Infra-red spectroscopy (IR)

The IR spectrum of synthesized nanoparticles was recorded using Fourier Transform Infra-Red spectrometer, Thermo Nicolet iS50 4000 cm⁻¹ to 100cm⁻¹ with resolution 0.2 cm⁻¹.

2.2.3.3 X-ray diffraction

The X-ray pattern was recorded using Bruker D8 Advance

2.2.4 APPLICATION

2.2.4.1 Dye adsorption of Eriochrome black T

About 0.01g of Eriochrome Black T was taken and made up to 100ml using distilled water. Then 15 ml of this solution was pipetted out into a beaker. The pipetted solution was made up to 100 ml using distilled water to make 15ppm Eriochrome Black T solution. 20 ml of the above solution was taken and 0.1 g of CuONP's were added. The solution was stirred for 1 hour and was centrifuged. The supernatant solution obtained was clear. The solution obtained after adding copper oxide nanoparticles as well as the coloured solution of Eriochrome Black T was given for UV-vis spectroscopy¹².

Chapter 3

Results and discussion

3.1 CHARACTERIZATION OF COPPER OXIDE NANOPARTICLES

3.1.1 INFRARED SPECTROSCOPY

The characteristic peaks corresponding to 486 cm^{-1} and 608 cm^{-1} in the FTIR spectra (fig 3.1) confirms the formation of copper oxide nanoparticles¹³.

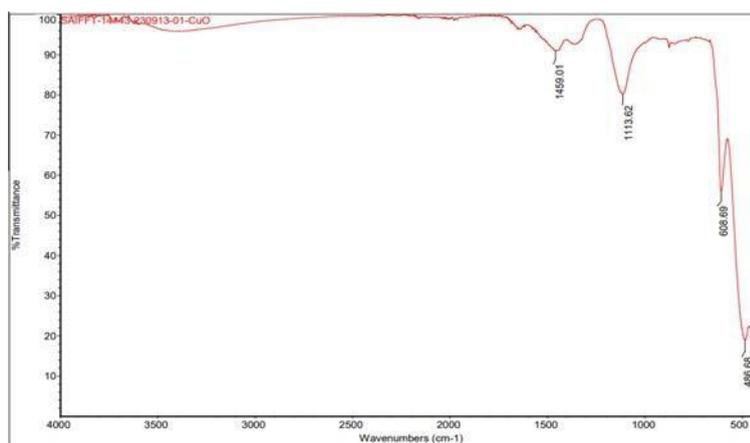


Fig 3.1 IR spectrum of copper oxide nanoparticles.

3.1.2 SEM-EDX

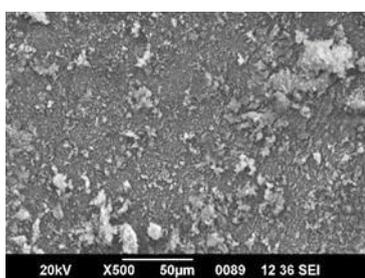


Fig 3.2

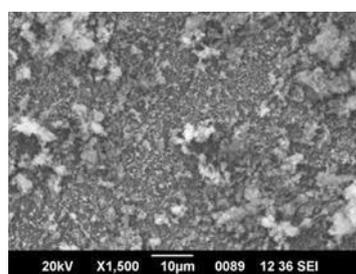


Fig3.3

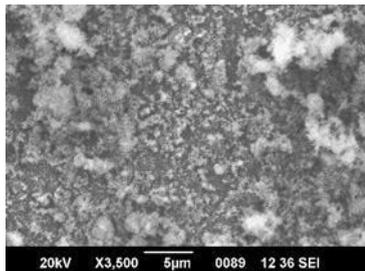


Fig 3.4

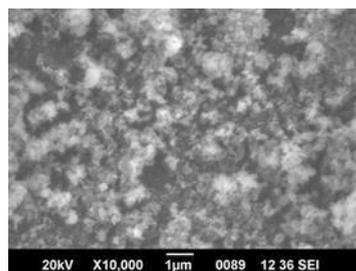


Fig 3.5

Scanning Electron Microscopy Analysis (SEM). SEM confirmed the morphology of the synthesized CuO nanoparticles. Figures shows the morphological form of CuONP's. From the SEM image, it is observed that the CuO nanoparticles are in highly collected form and have almost irregular morphology¹³.

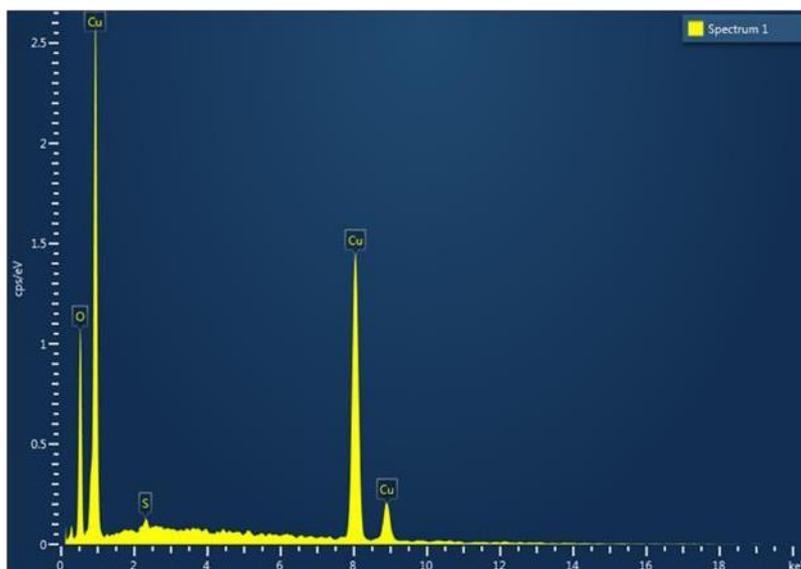


Fig 3.6 EDX spectrum of copper oxide nanoparticles.

Element	Line type	Weight%	Atomic %
O	K series	16.38	43.61
S	K series	0.48	0.64
Cu	K series	83.14	55.75
Total:		100	100

The EDX analysis revealed the chemical composition of the nanoparticles having atomic percent of 55.75 % for Cu, 43.61 % for O and 0.64 % for S. This shows the synthesized CuONP's are almost pure¹⁴.

3.1.3 XRD

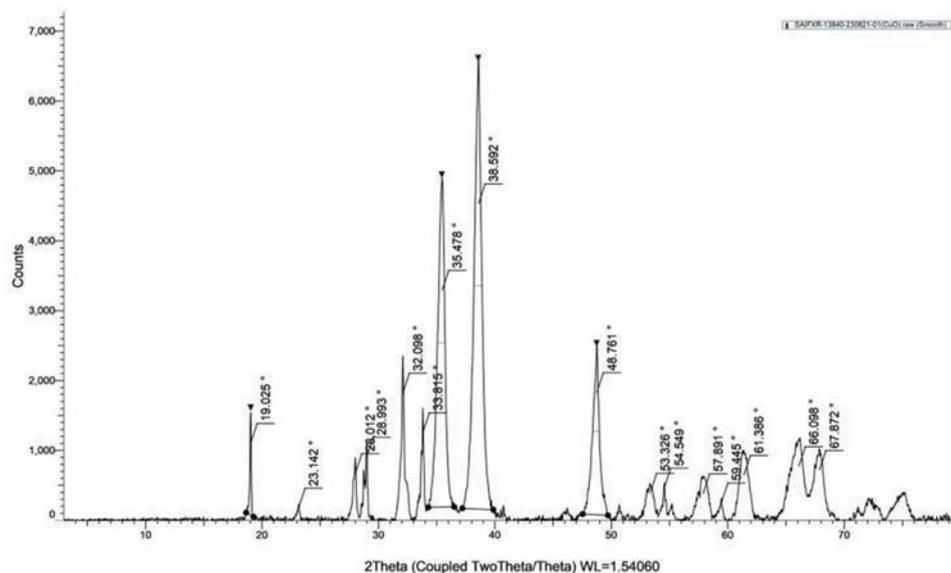


Fig 3.7 XRD pattern of copper oxide nanoparticles

The monoclinic crystal structure of CuO is represented by the sharp peaks observed at 2θ values of 32.098° , 35.478° , 38.592° , 48.761° , 53.326° , 61.386° , 67.872° respectively. These findings are consistent with prior

research. CuO is pure and well-arranged in a particular orientation, as shown by the highly crystalline and sharp diffraction peaks. Using Scherer formula, the crystalline size of the CuONP's can be calculated.

$$d = K\lambda / \beta \cdot \cos\theta$$

where λ is the X-ray radiation wavelength, β is the full-width half-maximum (FWHM), d is the average crystallite size, and K is the Scherer constant. The average size of CuONP's was calculated to be 27.661 nm. The size of CuONP's calculated is consistent with the earlier results¹⁵.

3.2 APPLICATION

3.2.1 ADSORPTION OF ERIOCHROME BLACK T



Fig 3.8 Eriochrome Black T before and after adsorption

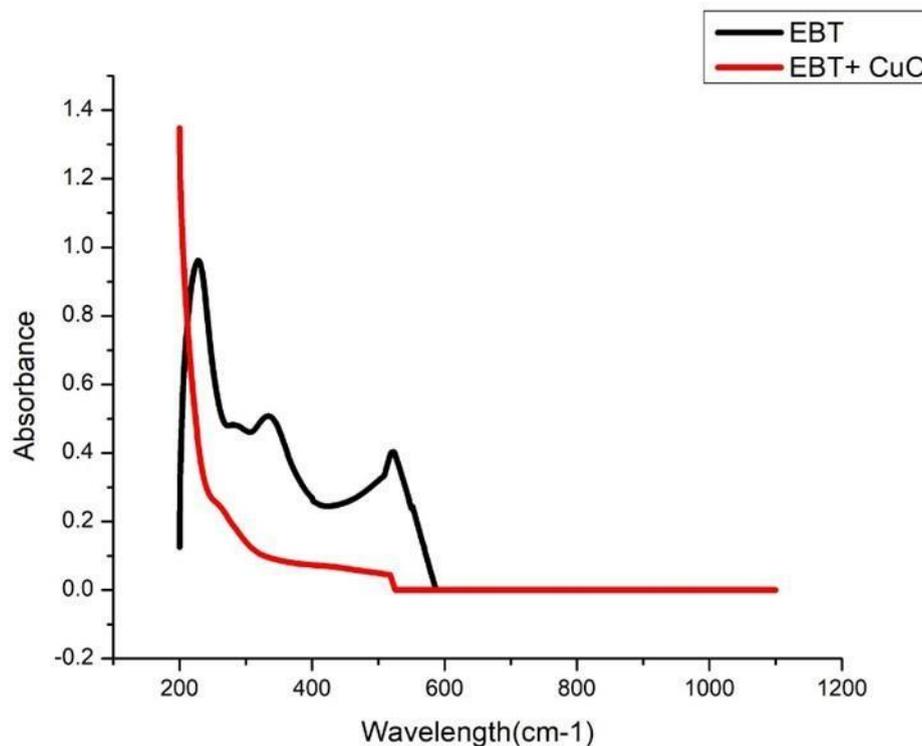


Fig 3.9 UV-visible spectrum of Eriochrome black T before and after adsorption.

Fig 3.8 shows the photograph of the before and after adsorption of EBT dye by CuO nanoparticles and fig 3.9 (graph) shows the absorbance spectrum of a EBT solution before and after adsorption. The spectrum shows a decline in the absorbance value after adding CuONP's. The decline in the absorbance value of the dye shows the complete adsorption of the dye by CuO nanoparticles.

Chapter 4

Conclusions

This study provides a distinctive approach for the green synthesis of CuONP's by using leaf extract of *Coleus amboinicus*. The Green synthesis method that takes advantage of using leaf extract from a medicinal plant is cost effective and eliminates the need for toxic chemical reducing agents. The prepared nanoparticles were characterized using, Infrared Spectroscopy FTIR, XRD and SEM-EDX. XRD and FTIR results corroborates the formation of CuONP's. The Sem images and EDX spectra provided an idea about the morphology and the elemental composition of the synthesized CuONP's respectively. The results shows that the CuONp's synthesized from *coleus amboinicus* leaf extract has ability to adsorb non- biodegradable dyes such as Eriochrome black T.

References

1. Mohamed EA. Green synthesis of copper & copper oxide nanoparticles using the extract of seedless dates. *Heliyon*. 2020;6(1). doi:10.1016/j.heliyon.2019.e03123
2. Dathar V. ANTIMICROBIAL, INSECTICIDAL POTENTIALS AND MEDICINAL PROPERTIES OF *Coleus Amboinicus*. <https://www.researchgate.net/publication/344271597>
3. Ayoman E, Hossini G, Haghghi N. Synthesis of CuO Nanoparticles and Study on Their Catalytic Properties. Vol 11.; 2015.
4. Wang F, Li H, Yuan Z, et al. A highly sensitive gas sensor based on CuO nanoparticles synthesized via a sol-gel method. *RSC Adv*. 2016;6(83):79343-79349. doi:10.1039/C6RA13876D
5. Wanninayake AP, Gunashekar S, Li S, Church BC, Abu-Zahra N. Performance enhancement of polymer solar cells using copper oxide nanoparticles. *Semicond Sci Technol*. 2015;30(6):064004. doi:10.1088/0268-1242/30/6/064004
6. Li Y, Wang X, Yang Q, et al. Ultra-fine CuO Nanoparticles Embedded in Three-dimensional Graphene Network Nano-structure for High-performance Flexible Supercapacitors. *Electrochim Acta*. 2017;234:63-70. doi:10.1016/j.electacta.2017.02.167
7. Ren G, Hu D, Cheng EWC, Vargas-Reus MA, Reip P, Allaker RP. Characterisation of copper oxide nanoparticles for antimicrobial applications. *Int J Antimicrob Agents*. 2009;33(6):587-590. doi:10.1016/j.ijantimicag.2008.12.004
8. Sibhatu AK, Weldegebrieal GK, Sagadevan S, Tran NN, Hessel V. Photocatalytic activity of CuO nanoparticles for organic and inorganic pollutants removal in wastewater remediation. *Chemosphere*. 2022;300:134623. doi:10.1016/j.chemosphere.2022.134623

9. MH, Liu HS, Tai CY. Preparation of copper oxide nanoparticles and its application in nanofluid. *Powder Technol.* 2011;207(1-3):378-386. doi:10.1016/j.powtec.2010.11.022
10. Sivayogam D, Kartharinal Punithavathy I, Johnson Jayakumar S, Mahendran N. Study on structural, electro-optical and optoelectronics properties of CuO nanoparticles synthesis via sol gel method. *Mater Today Proc.* 2022;48:508-513. doi:10.1016/j.matpr.2021.04.494
11. Kumar PPNV, Shameem U, Kollu P, Kalyani RL, Pammi SVN. Green Synthesis of Copper Oxide Nanoparticles Using Aloe vera Leaf Extract and Its Antibacterial Activity Against Fish Bacterial Pathogens. *Bionanoscience.* 2015;5(3):135-139. doi:10.1007/s12668-015-0171-z
12. Narayanan M, Salmen SH, Chinnathambi A, et al. Nanoparticle-assisted removal of EBT dye from textile wastewater: Towards sustainable green gram seedling cultivation. *J Taiwan Inst Chem Eng.* Published online December 2023:105258. doi:10.1016/j.jtice.2023.105258
13. Ramzan M, Obodo RM, Mukhtar S, Ilyas SZ, Aziz F, Thovhogi N. Green synthesis of copper oxide nanoparticles using Cedrus deodara aqueous extract for antibacterial activity. In: *Materials Today: Proceedings.* Vol 36. Elsevier Ltd; 2019:576-581. doi:10.1016/j.matpr.2020.05.472
14. S. Prabhua b *, TDTPVB PonK. Investigation on the Photocatalytic and Antibacterial Activities of Green synthesized Cupric Oxide Nanoparticles using Clitoria ternatea .
15. Padil, V. V. T., & Černík, M. (2013). Green synthesis of copper oxide nanoparticles using gum karaya as a biotemplate and their antibacterial application. *International Journal of Nanomedicine*, 8, 889–898. <https://doi.org/10.2147/IJN.S40599>
16. Rabiee, N., Bagherzadeh, M., Kiani, M., Ghadiri, A. M., Etessamifar, F., Jaberizadeh, A. H., & Shakeri, A. (2020). Biosynthesis of copper oxide nanoparticles with potential biomedical applications. *International Journal of Nanomedicine*, 15, 3983– 3999. <https://doi.org/10.2147/IJN.S255398>

17. Ramzan, M., Obodo, R. M., Mukhtar, S., Ilyas, S. Z., Aziz, F., & Thovhogi, N. (2019). Green synthesis of copper oxide nanoparticles using *Cedrus deodara* aqueous extract for antibacterial activity. *Materials Today: Proceedings*, 36, 576–581.
<https://doi.org/10.1016/j.matpr.2020.05.472>
18. Rashad, M., & Al-Aoh, H. A. (2019). Promising adsorption studies of bromophenol blue using copper oxide nanoparticles. *Desalination and Water Treatment*, 139, 360–368.
<https://doi.org/10.5004/dwt.2019.23296>
19. Rather, M. Y., & Sundarapandian, S. (2022a). Facile Green Synthesis of Copper Oxide Nanoparticles and Their Rhodamine-b Dye Adsorption Property. *Journal of Cluster Science*, 33(3), 925–933.
<https://doi.org/10.1007/s10876-021-02025-4>
20. Ren, G., Hu, D., Cheng, E. W. C., Vargas-Reus, M. A., Reip, P., & Allaker, R. P. (2009). Characterisation of copper oxide nanoparticles for antimicrobial applications. *International Journal of Antimicrobial Agents*, 33(6), 587–590.
<https://doi.org/10.1016/j.ijantimicag.2008.12.004>
21. Srivastava, V., & Choubey, A. K. (2021). Investigation of adsorption of organic dyes present in wastewater using chitosan beads immobilized with biofabricated CuO nanoparticles. *Journal of Molecular Structure*, 1242.
22. Sukumar, S., Rudrasenan, A., & Padmanabhan Nambiar, D. (2020). Green-Synthesized Rice-Shaped Copper Oxide Nanoparticles Using *Caesalpinia bonducella* Seed Extract and Their Applications. *ACS Omega*, 5(2), 1040–1051.
23. Tran, T. H., & Nguyen, V. T. (2014). Copper Oxide Nanomaterials Prepared by Solution Methods, Some Properties, and Potential

- Applications: A Brief Review. *International Scholarly Research Notices*, 2014, 1–14. <https://doi.org/10.1155/2014/856592>
24. Wongpisutpaisan, N., Charoonsuk, P., Vittayakorn, N., & Pecharapa, W. (2011). Sonochemical synthesis and characterization of copper oxide nanoparticles. *Energy Procedia*, 9, 404–409. <https://doi.org/10.1016/j.egypro.2011.09.044>.
25. Kumar, P. P. N. V., Shameem, U., Kollu, P., Kalyani, R. L., & Pammi, S. V. N. (2015b). Green Synthesis of Copper Oxide Nanoparticles Using Aloe vera Leaf Extract and Its Antibacterial Activity Against Fish Bacterial Pathogens. *BioNanoScience*, 5(3), 135–139. <https://doi.org/10.1007/s12668-015-0171-z>
26. Lanje, A. S., Sharma, S. J., Pode, R. B., & Ningthoujam, R. S. (n.d.). Synthesis and optical characterization of copper oxide nanoparticles. www.pelagiaresearchlibrary.com
27. Manyasree, D., Peddi, K. M., & Ravikumar, R. (2017). CuO nanoparticles: Synthesis, characterization and their bactericidal efficacy. *International Journal of Applied Pharmaceutics*, 9(6), 71–76. <https://doi.org/10.22159/ijap.2017v9i6.71757>.
28. Amin, F., Fozia, Khattak, B., Alotaibi, A., Qasim, M., Ahmad, I., Ullah, R., Bourhia, M., Gul, A., Zahoor, S., & Ahmad, R. (2021). Green Synthesis of Copper Oxide Nanoparticles Using *Aerva javanica* Leaf Extract and Their Characterization and Investigation of in Vitro Antimicrobial Potential and Cytotoxic Activities. *Evidence-Based Complementary and Alternative Medicine*, 2021. <https://doi.org/10.1155/2021/5589703>
29. Abboud, Y., Saffaj, T., Chagraoui, A., El Bouari, A., Brouzi, K., Tanane, O., & Ihssane, B. (2014). Biosynthesis, characterization and antimicrobial activity of copper oxide nanoparticles (CONPs) produced using brown alga extract (*Bifurcaria bifurcata*). *Applied Nanoscience (Switzerland)*, 4(5), 571–576. <https://doi.org/10.1007/s13204-013-0233-x>