PROJECT REPORT

ON

“DYE ADSORPTION STUDIES AND ANTIBACTERIAL EFFICIENCY OF NANOCURCUMIN FUNCTIONALISED COPPER OXIDE NANOPARTICLES”

Submitted by
LAKSHMI K K
AM21CHE009

In partial fulfillment for the award of the
Post Graduate Degree in Chemistry

DEPARTMENT OF CHEMISTRY
AND
CENTRE FOR RESEARCH

ST. TERESA’S COLLEGE (AUTONOMOUS)
ERNAKULAM

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M.Sc. CHEMISTRY PROJECT REPORT

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This is to certify that the project **"Dye Adsorption Studies and Antibacterial Efficiency of Nanocurcumin Functionalised Copper Oxide Nanoparticles"** is the work done by **Lakshmi K K.**

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Submitted to the Examination of Master's degree in Chemistry

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DEPARTMENT OF CHEMISTRY
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ERNAKULAM

CERTIFICATE

This is to certify that the project work entitled "**Dye Adsorption Studies and Antibacterial Efficiency of Nanocurcumin Functionalised Copper Oxide Nanoparticles**" is the work done by **Lakshmi K K** under the guidance of **Dr.Ambili K S,** Assistant Professor, Department of Chemistry and Centre for Research, St. Teresa's College, Ernakulam in partial fulfilment of the award of the Degree of Master of Science in Chemistry at St. Teresa's College, Ernakulam affiliated to Mahatma Gandhi University, Kottayam.

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DECLARATION

I hereby declare that the project work entitled “**Dye Adsorption Studies and Antibacterial Efficiency of Nanocurcumin Functionalised Copper Oxide Nanoparticles**” submitted to Department of Chemistry and Centre for Research, St. Teresa’s College (Autonomous) affiliated to Mahatma Gandhi University, Kottayam, Kerala is a record of an original work done by us under the guidance of **Dr.Ambili K S**, Assistant Professor, Department of Chemistry and Centre for Research, St. Teresa’s College (Autonomous), Ernakulam. This project work is submitted in the partial fulfillment of the requirements for the award of the Degree of Master of Science in Chemistry.

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Chapter1

Introduction

1.1 WATER POLLUTION

Over 96.5 percent of the water on the globe is found in the seas, which also cover 71 percent of its surface [1]. Water pollution is any kind of contamination of water bodies, such as rivers, lakes, or wetlands, with pollutants that pose a risk to the environment or public health. Such pollution, particularly in impoverished countries, is a major cause of mortality and disease globally. Many lakes and rivers are polluted, even in more developed countries where piped water sources ensure that water pollution offers less of a direct hazard to human health [2]. The impacts of colour in water streams on daily life are not very noteworthy (Fig.1.1& 1.2). With an output of 1,000,000 tonnes worldwide, dyes are used in small- and large-scale businesses worldwide, including tanneries, culinary, cosmetic, textile, and pharmaceutical sectors. The textile industry is primarily responsible for a significant portion of the ecosystem's dye emissions. Only declining industries release about 7.5 metric tonnes annually [3].To decrease its detrimental effects is the best way to stop widespread water contamination. Before being dumped in water bodies, rubbish must be treated to reduce large-scale water contamination. This effluent can be reused in agriculture or other sectors by reducing its dangerous level [4].

Figure 1.1Rivers dying due to dyeing

Figure 1.2 pollution in water bodies by textile industries

1.1.1 TEXTILE DYES

The mills produce millions of litres of this hazardous toxic effluent, which is rich in colour and organic compounds from dyeing and finishing salts. The presence of heavy metals including copper, arsenic, lead, cadmium, mercury, nickel, and cobalt as well as sulphur, naphthol, vat dyes, nitrates, acetic acid, soaps, and other auxiliary chemicals renders the effluent particularly hazardous. Hazardous water additives include hydrocarbon-based softeners, non-biodegradable dyeing chemicals, and formaldehyde-based colour fixing agents. The mill wastewater also commonly has high pH and temperature levels, both of which are quite dangerous. Along with adding colour and oily scum, colloidal debris raises the turbidity of the water, making it harder to see and smell. Photosynthesis depends on sunlight, which is blocked by this object [5].

1.1.2 WATER POLLUTION FROM TEXTILE INDUSTRY

One of the most significant sources of pollution is waste water from the dyeing of textiles. It is acknowledged as a major contributor to environmental contamination. High levels of textile days in water bodies disrupt biological activity in aquatic life as well as the photosynthetic process of aquatic plants or algae by stopping the reoxygenation capability of the receiving water and cutting off sunlight. The toxic effects of dyes due to their long-term presence in the environment, accumulation in sediments, particularly in fish or other aquatic life forms, decomposition of pollutants into carcinogenic or mutagenic compounds, as well as low acrobic biodegradability, can also have a negative impact on the aquatic environment. To effectively and economically treat the waste water from textile dyeing, a number of techniques have been developed. There is still no universal standard, despite the strict emissions rules that several countries have established. The reduction of waste is essential for bringing down production costs and pollution levels.

1.1.3 TREATMENT OF TEXTILE DYES IN WASTE WATER

The wastewater from the textile manufacturing industry has been treated using a variety of techniques, including physical, chemical, biological, integrated treatment procedures, and other technologies. The waste water from textile dyeing can usually be treated using these processes pretty effectively.

1.1.3.1 CHEMICAL TREATMENT PROCESS

The oxidative process includes the use of Fentons reagent, ozonation, photochemicals, solium hypochloride, cucurbituril, and electrochemical degradation. The most used oxidative techniques for chemical decolorization. This is mostly due to how simply it may be used.

1.1.3.2 PHYSICAL TREATMENT PROCESS

As a method of treating aqueous effluent, adsorption techniques are becoming more and more used since they are efficient at eliminating contaminants that are too stable for conventional methods and are also viable from an economic aspect. Natural clay, agricultural waste products, fly ash, wood chips, silica gel, membrane filtration, ion exchange irradiation and electrokinetic coagulation are some of the materials employed in the physical process.

1.1.3.3 BIOLOGICAL TREATMENT PROCESS

An approach that is pollution-free and excellent for the environment is biological treatment, which makes use of a variety of potential organisms. The biological process entails the use of an anaerobic fluorescent dye bioremediation system, further microbial culture, white-rot fungi to remove colour, and living and dead microbial biomass for adsorption.

1.1.4 NANOTECHNOLOGY IN WASTE WATER TREATMENT

Nanotechnology is one of the top five cutting-edge technologies being used to develop water filtration systems. This is as a result of its wide variety of applications in the detection, remediation, and prevention of water contamination. The unique qualities of nanomaterials, such as their enormous surface area, photosensitivity, catalytic and antibacterial activity, electrochemical, optical, and magnetic capabilities, as well as their changeable pore size and surface chemistry, enable a wide range of applications. Nanoparticles (NPs) are tiny particles with sizes between 1 and 100 nm that are essential for removing impurities from aqueous systems.They are composed of metals and metal oxides, such as titanium dioxide, metal oxide nanomaterial, cupric oxide, etc., and can be coupled with an aqueous suspension.

Although many technologies have long employed structures with one or more nanoscale dimensions accidentally and there are many examples of them in the natural world, it has only recently become practical to do so on purpose. Numerous applications of nanotechnology involve innovative materials with radically different properties and results from the same materials generated at larger scales. This is due to the exceptionally high surface to volume ratio of nanoparticles compared to larger particles, as well as impacts that are observable at that small scale but not at higher scales. Applications of nanotechnology have the potential to be exceedingly beneficial and have a significant impact on society. Nanotechnology has already been adopted by the information and communications sectors as well as the food and energy sectors. It is also used in a number of pharmaceuticals and medical items. Additionally, the use of nanomaterials may open up new avenues for reducing environmental contamination. Every time a brand-new danger is thought to exist, a careful analysis of that risk's characteristics is necessary. The risk management practises can then, if necessary, be altered by this analysis[7]. There are some phenomena that become more pronounced as the system gets smaller. These include statistical and quantum mechanical phenomena, such as the "quantum size effect," which changes the electrical properties of materials when particle size is significantly reduced. Changing the dimensions from macro to micro has no influence on this result. However, quantum effects can become significant when the nanoscale size range is reached, frequently at distances of 100 nanometers or less, the so-called quantum domain. A number of physical (mechanical, electrical, optical, etc.) characteristics also change when compared to macroscopic system. One illustration is how changing the surface area to volume ratio can change a material's mechanical, thermal, and catalytic properties. Nanoionics is the broad term for nanoscale diffusion and reactions, nanostructured materials, and nanodevices with quick ion transport. The mechanical properties of nanosystems are a focus of nanomechanics research. The catalytic activity of nanomaterials makes interactions with biomaterials potentially hazardous. The ability of materials to exhibit different properties at the nanoscale than they do at the macroscale allows for novel applications. For instance, stable materials like aluminium may become combustible, opaque materials like copper could turn translucent, and insoluble minerals like gold could change their solubility. Gold, which is chemically inert at larger scales, can function as a potent chemical catalyst at nanoscales. A significant source of the fascination with nanotechnology is the quantum and surface phenomena that matter exhibits at the nanoscale[8].

1.2 NANOMATERIALS

Nanomaterials (NMs) chemistry is a relatively recent field of research. Many different nanotechnological gadgets are built on NMs. Even if NMs have always existed in nature, scientists a few decades ago would have been baffled. Particles between 1 and 100 nm in size and with at least one three-dimensional dimension are referred to as "nanomaterials". In terms of their chemical and physical properties, molecules or atoms in NMs states behave differently from the comparable materials in bulk. NMs show quantum phenomena and have a larger surface area because of their incredibly small size. As a result, NMs' unique properties cannot be predicted by comparing them to bulk materials. Advances in the realm of nanotechnology have encouraged the design and creation of novel NMs with different sizes and morphologies for multiple interdisciplinary applications in material science, chemistry, and biology as well as allied domains. To produce NMs, a variety of synthesis techniques can be employed. NMs are made up of a variety of components, including as metals, metal-organic frameworks (MOF), carbon dots, polymer nanoparticles, and others. Additionally, NMs can be found in a wide range of morphological geometries, including disc, cube, sphere, and rod-like forms. Synthetic NMs sometimes require changing their surfaces in order to stabilise them because of their nano size, which results in significant chemical reactivity and inhibits them from aggregating. The NMs can also be surface functionalized to accommodate any necessary applications. Sensing and bioimaging are essential in biological and diagnostic applications (Fig. 1.3). Therefore, the best materials for advancing science and solving puzzles are luminous NMs[9].

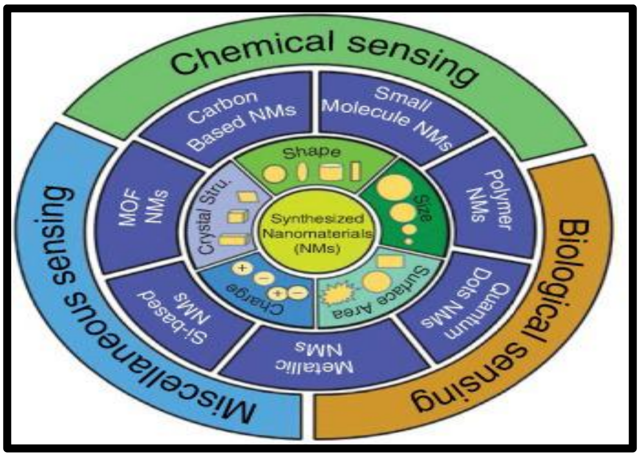


Figure 1.3 Applications of nanomaterials in sensing

We will discuss a few instances of nanomaterials below in order to better grasp their properties. We'll find that while certain nanoparticles' behaviour is well understood, others pose more difficulties. One-dimensional nanomaterials like thin films and customised surfaces have been developed and used for decades in fields like the manufacture of electrical devices, chemistry, and engineering. For instance, thin films are necessary for the operation of many devices in the silicon integrated circuit industry, and it is usual practise to control film thicknesses that are near to the atomic level. Layers that are only one atom or molecule deep, or monolayers, are often made and used in chemistry. The most notable example of this new class of materials is graphene. Even with extremely complex layers (such lubricants and nanocoatings), the atomic level upwards of these layers' formation and properties is relatively well understood. Advances are being made in the control of surface composition, surface smoothness, and film growth. Engineered surfaces with specialised properties are frequently used in a number of applications, including catalysts and fuel cells. These properties could include a large surface area or a specific reactivity. All of these applications may benefit from the vast surface area that nanoparticles offer and their capacity to self-assemble on a support surface[10].

1.3 CLASSIFICATION OF NANOPARTICLES

Nanoparticles can be classed based on their size, shape, physical attributes, and chemical makeup. The classification of these nanoparticles typically dictates their function**.**

1.3.1 CARBON-BASED NANOPARTICLES

Carbon nanotubes (CNTs) and fullerenes make up the majority of carbon-based nanoparticles. Given that they are 100 times stronger than steel, the usage of these nanoparticles usually concentrates on structural reinforcement.Single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) are two different types of CNTs. Because they are non-conductive across the tube yet thermally conductive along its length, CNTs are special in a way.Carbon allotropes known as fullerenes have hollow cage structures made up of sixty or more carbon atoms. The hollow football-like Buckminster fullerenes structure of C60. These carbon units are pentagonal and hexagonal in shape. These beneficial carbon-based nanoparticles have a structure, high strength, and electron affinity in addition to electrical conductivity.

1.3.2 CERAMIC NANOPARTICLES

These inorganic solid nanoparticles are composed of phosphates, carbonates, carbides, and oxides. Because of their excellent heat resistance and chemical inertness, ceramic nanoparticles are utilised in biological imaging, medication administration, photocatalysis, and dye photodegradation.These nanoparticles' specific features, such as size, surface area, porosity, and surface-to-volume ratio, may be regulated, making them effective as drug delivery devices.Medications for conditions including cancer, glaucoma, and bacterial infections have all been successfully transported using ceramic nanoparticles.

1.3.3 METAL NANOPARTICLES

Metal nanoparticles can be generated chemically, electrochemically, or photochemically from metal precursors. Chemical methods are used to create metal nanoparticles by chemically reducing the metal-ion precursors in solution. The resultant nanostructures can adsorb small molecules and have a high surface energy.Several scientific fields, including biomolecular imaging and detection as well as environmental and analytical applications, use metal nanoparticles. For example, gold nanoparticles are used to coat the sample before SEM analysis in order to enhance SEM and produce superb electron microscopy images.

1.3.4 SEMICONDUCTOR NANOPARTICLES

Semiconductor nanoparticles can be found in the periodic table's groups II–VI, III–V, or IV–VI and exhibit characteristics that are similar to both those of metals and non-metals. These nanoparticles' wide bandgaps make it possible to modify a variety of features. Examples of semiconductor nanoparticles include GaN, GaP, InP, and InAs from groups III to V, as well as ZnO, ZnS, CdS, CdSe, and CdTe from groups II to VI, silicon, and germanium from group IV.Electronics, photocatalysis, nanophotonics, and water splitting are some uses for semiconductor nanoparticles[11].

1.4 SYNTHESIS OF NANOPARTICLES

A structure in the 1-100 nm range with at least one or two dimensions is referred to as a nanostructure. Nanoparticles can be produced in a variety of ways 122 Synthesis of Inorganic Nanomaterials. Both dry and liquid-dispersed nanoparticles can be produced using these techniques. Nanostructures can be produced by starting with atoms or by shrinking microparticles to nanoparticles**.**

1.4.1 COPRECIPITATION

When nucleation, growth, coarsening, and/or agglomeration processes occur simultaneously, coprecipitation reactions result. Coprecipitation reactions exhibit the following characteristics:

- An important process is nucleation, during which numerous small particles are made.
- The by-products are frequently intractable compounds produced in extremely saturated conditions.
- The size, shape, and characteristics of the products are significantly impacted by secondary processes such Ostwald ripening and aggregation.

Common methods of synthetic coprecipitation include:

- Metal chalconides are created by the reactions of molecular precursors, and oxides are created from both aqueous and non-aqueous solutions.
- Metals generated from aqueous solutions through reduction from non-aqueous solutions, electrochemical reduction, and breakdown of metallorganic precursors.
- Microwave/sonication-assisted coprecipitation.

1.4.2 HYDROTHERMAL TECHNIQUE

The hydrothermal approach received the most praise from experts in several scientific fields. Hydrothermal synthesis has been used to effectively produce a number of significant solids, including microporous crystals, superionic conductors, chemical sensing oxides, electronically conducting solids, complex oxide ceramics, fluorides, magnetic materials, and luminescence phosphors. The term "hydrothermal synthesis" refers to the procedure of producing compounds by chemical reactions in a sealed, heated solution that is above the ambient pressure and temperature. Most scientists and technologists from other areas praised the hydrothermal process. Hydrothermal synthesis has been used to effectively produce a number of significant solids, including magnetic materials, luminescence phosphors, superionic conductors, chemical sensing oxides, electronically conducting solids, complex oxide ceramics, and microporous crystals. The term "hydrothermal synthesis" refers to the process of generating compounds by chemical reactions in a sealed, heated solution that is above the ambient pressure and temperature.

1.4.3 SPUTTERING

Atoms are ejected from the surface of a material (the target) during the sputtering process by being blasted with high-energy particles. During the momentum transfer procedure called sputtering, atoms from a cathode or target are driven away by bombarding ions. Sputtered atoms travel until they strike a substrate, at which point they deposit to form the required layer.The simplest source of ions for sputtering is the well-known phenomenon of glow discharge, which is brought on by an applied electric field between two electrodes in a gas at low pressure.The gas deteriorates and starts to conduct electricity when a specified minimum voltage is reached. Such an ionised gas is known as plasma. Plasma ionisation is accelerated at the target by a strong electric field. When the plasma ions strike the target, their electrical charge is neutralised, and they rejoin the process as atoms. The neutralisation process leaves the target surface positively charged if the target is an insulator. It's possible that this charge may eventually stop repelling bombarding ions and the sputtering process will stop. To take enough electrons from the plasma to complete the procedure and discharge the surface charge, the polarity must be reversed. As demonstrated in Figure 126 of Synthesis of Inorganic Nanomaterials, an automatic periodic polarity reversal takes place when a radio-frequency

(RF) voltage is applied to the target assembly. As a result, this type of sputtering is called "RF sputtering."

1.4.4 MICROEMULSION

Even though the microemulsion technique is one of the greatest ways to make inorganic nanoparticles, it is still unclear how nanoparticles grow in microemulsions. However, a process for the production of nanoparticles in microemulsions has been put forth by certain scientists. After combining the reactant-containing microemulsion material, reactant exchange takes place during the collision of water droplets in the microemulsion. A precipitation reaction happens in the nanodroplets as a result of the reactant exchange taking place too quickly. Following this precipitation reaction, the primary particles expand and coagulate, which creates the final nanoparticles that are stabilised by surfactants and/or are encircled by water.

1.4.5 MICROWAVE ASSISSTED SYNTHESIS

The use of microwaves in synthesis is frequently used in biological and nanotechnology processes. With higher yields and fewer byproducts, chemical reactions usually occur faster than with traditional convection heating approaches. Microwave reactors provide great control over reaction mixing in addition to withstanding high temperatures and pressures and demonstrating repeatability from reaction to reaction. Microwave-assisted methods provide superior technical control over the division of the nucleation and growth stages of nanomaterial creation when the reaction is initiated at room temperature. With microwave-assisted heating, selectivity in the activation of the precursor materials for nanomaterials might be feasible for scaling. Microwave synthesis can selectively heat either the solvent or the precursor molecules to produce nanomaterials.

1.4.6 LASER ABLATION

The technique of laser ablation involves removing material from a solid surface by exposing it to a laser beam. Low laser flux heats the substance, which then evaporates or sublimates as a result of the absorbed laser energy. Typically, the material undergoes plasma formation at high laser flux. If the laser intensity is high enough, it is also feasible to ablate material using a continuous wave laser beam. However, the phrase "laser ablation" normally refers to the removal of material using a pulsed laser.

1.4.7 SOL-GEL PROCESS

Sol-gel is a technique for creating solid materials out of small molecules. An ultimate diphasic system with a liquid phase and a solid phase, with morphologies ranging from discrete particles to continuous polymer networks, arises from the sol (or solution) in this chemical reaction.Ceramic powders that are evenly and finely powdered can be produced by precipitation. These powders with single- and multi-component compositions can be produced using nanoscale-sized particles for dentistry and biomedical purposes. To make a clear and stable sol solution, combine the raw materials equally in the liquid phase and use the hydrolysis and polycondensation processes. Materials with microstructures and even nanostructures can be produced through a variety of processes, including drying and sintering. After sols have aged, colloidal particles slowly aggregate to create gels with three-dimensional network structures.

1.4.8 ULTRASOUND

Ultrasound has become a more popular method for creating nanoparticles. When liquids are exposed to ultrasonic energy, ultrasonic cavitation will form. In the face of challenging conditions, ultrasonic cavitation produces a variety of physical and chemical consequences, such as high temperature, pressure, and cooling rate, which creates a unique environment for chemical reactions. Ultrasound is a fantastic method for producing nanoparticles with customizable morphologies.

Advantages:

- Nanoparticle production takes place at ambient temperature.
- Nanoparticle production is faster thanks to ultrasound technology's surprisingly fast reaction rates.
- The procedure is quick and simple.

Some examples of the various configurations and assemblies of nanoparticles include nanobelts, nanorings, core-in-shell, and cluster-in-cluster bimetallic nanoparticles.

1.4.9 SPARK DISCHARGE

Spark discharge nanoparticle synthesis is a promising method for low-cost industrial scale nanofabrication of novel materials. An electric spark is an abrupt electrical discharge that occurs when an ionised, electrically conductive channel forms through a normally insulating medium, most often air or other gases or gas combinations.

1.4.10 TEMPLATE SYNTHESIS

The need for a quick, environmentally friendly method of nanoparticle synthesis is growing as green chemistry receives greater attention. One of the most promising methods for producing monodispersed inorganic nanoparticles has been called template synthesis, which utilises the uniform void spaces of porous materials as hosts to confine the synthesised nanoparticles as guests. The template accomplishes two tasks. It acts as a framework for organising a device's numerous processes, active parts, and interfaces. It first makes it feasible to reproduce the structure with the highest degree of reproducibility[12].

1.5 GREEN SYNTHESIS OF NANOPARTICLES

The three main prerequisites for the synthesis of nanoparticles are the selection of an eco-friendly or green solvent, a good reducing agent, and a secure stabilising material. Nanoparticles have been produced using a variety of synthetic processes, including physical, chemical, and biosynthetic processes. Generally speaking, the chemical procedures used are very expensive and contain hazardous and dangerous materials that are to blame for a number of environmental issues. The biosynthetic pathway provides a safe, biocompatible, and green way to make nanoparticles for biomedical applications. This synthesis can involve fungi, algae, bacteria, plants, and other living things. Because they are simple to use, efficient, cost-effective, and practical, plant extract-based biosynthetic techniques have gained increased attention as a great alternative to conventional preparation procedures for the synthesis of nanoparticles.Heavy metals have the ability to build up in different areas of plants in varying amounts. As a result, plant extract-based biosynthetic approaches have drawn more attention as an easy, effective, affordable, and doable way as well as a great substitute for traditional preparation methods for nanoparticle manufacturing. Heavy metals have the ability to build up in different areas of plants in varying amounts. As a result, plant extract-based biosynthetic approaches have drawn more attention as an easy, effective, affordable, and doable way as well as a great substitute for traditional preparation methods for nanoparticle manufacturing.In a "one-pot" production process, the metallic nanoparticles can be reduced and stabilised using a variety of plants. To further explore their various applications, numerous researchers have developed metal/metal oxide nanoparticles utilising a green synthesis technique employing plant leaf extracts[14].

1.6 APPLICATION OF NANOPARTICLES

1.6.1 NANOMEDICINE

Heavy metals have the ability to build up in different areas of plants in varying amounts. As a result, plant extract-based biosynthetic approaches have drawn more attention as an easy, effective, affordable, and doable way as well as a great substitute for traditional preparation methods for nanoparticle manufacturing.The term "nanomedicine" references to medical applications of nanotechnology. It addresses topics like biological devices, nanoscaled biosensors, and nanomaterials utilised in medicine. Future biological nanoscaled machine applications are anticipated. Nanomaterials could be dangerous, and the impact they have on the environment is quite concerning.

Nanomaterials can be altered to perform a wide range of specialised functions. This can be achieved by mixing the nanomaterials with additional proteins or structures. The nanomaterials are comparable in size to various biomolecules and structures, making them suitable for both in vitro and in vivo biological research and applications. To date, the intersection of nanomaterials and biology has produced a wide range of diagnostic tools, contrast agents, analytical tools, physical therapy applications, drug delivery vehicles, etc.

Improved disease diagnosis, early detection, and prevention measures are supported by nanomedicine. Gene sequencing has become simpler because to the advent of nanotechnology, which produced gold nanoparticles as one example. They are also used to find genetic sequences when coupled to the short DNA fragments. It is possible to reproduce or repair damaged tissue using nanotechnology.Organ transplantation and artificial implantation could both be revolutionised by nanotechnology. It has been shown that magnetic nanoparticles may be used to successfully isolate and group stem cells. Quantum dots, on the other hand, have been used in molecular imaging, stem cell tracing, etc. The proliferation and differentiation of stem cells can be managed by utilising specifically made nanoparticles51. Another benefit of nanotechnology is the regeneration and neuroprotection of the Central Nervous System. Parkinson's disease is one of the most well-known neurodegenerative conditions. Using an intracranial nano-enabled scaffold device (NESD) for site-specific dopamine administration to the brain is a fantastic technique to reduce the side effects of Parkinson's disease treatment on the periphery. Examples of novel strategies include the use of peptides and peptidic nanoparticles as creative remedies for various CNS disorders and the activation of signalling signals for regulated axon growth. They can also bring damaged neurons back to full health in order to safeguard the brain and make it easier for chemicals and medications to pass over the blood brain barrier. The majority of amyloid beta plaques are found in the brains of people with Alzheimer's disease. These nanoparticles may reduce the plaques because of their strong affinity for them, reducing the signs and symptoms of Alzheimer's disease. Tuberculosis is an infectious disease that can be lethal.More effective and reasonably priced TB pharmacotherapy is now possible because to recent developments in nano-based drug delivery technology for the encapsulation and release of antiTB drugs52. Nano filled composite resin materials provide good wear resistance, hardness, and great aesthetic values on par with operational dentistry because of their exceptional lustre retention and polishability. Spherical silicon dioxide nano fillers have the capacity to alter the amount of inorganic phase present when used in active dentistry.

1.6.2 DRUG DELIVERY

One of the most well-known applications of nanotechnology is the targeted medicine delivery to certain cells. Because medication can be delivered directly to the affected location, drug consumption can be decreased. Furthermore, this decreases the side effects of the drugs. Targeted drug administration is essential for lowering drug side effects and treatment costs by limiting drug consumption. The goal of medication delivery is to make the drug more bioavailable at the intended site in the body and more readily available for a defined period of time. Nanoengineered devices can be used for this since they can target particular molecules. Reduced invasiveness, fast biochemical reactions, and the ability to implant inside the body are only a few advantages of nano sized technologies. These devices are quicker and more effective than conventional drug delivery techniques.

The efficiency of nanomedicine depends on:

(a) efficient drug encapsulation
(b) successful transport of the drug to specific site, and
(c) efficient release of drug.

1.6. 3 CANCER TREATMENT

Due to their extremely high surface area to volume ratios, nanoparticles can attach various functional groups to them, enabling them to find and bind to specific cancer cells. Furthermore, nanoparticles (10-100 nm) preferentially accumulate in cancer sites due to the absence of a functioning lymphatic drainage system in tumours. Drug resistance, a lack of selectivity, and solubility are some of the problems of conventional cancer chemotherapy that can be avoided by using nanoparticles.

1.6.4 IMAGING

Imaging Nanoparticles have a huge promise as tools and approaches for in vivo imaging. When utilising nanoparticle-based contrast agents, images (like ultrasound images) can have a beneficial distribution and enhanced contrast. Nanoparticles can help with the imaging of many stages of cardiovascular diseases, such as blood clotting, angiogenesis, atherosclerosis, etc. Because of their small size, nanoparticles can be very useful in oncology, especially in imaging. Magnetic resonance imaging (MRI) using QDs results in amazing images of cancer regions. Cadmium selenide nanoparticles glitter when exposed to UV light. When injected, they penetrate the cancer cells and draw attention to them. Nanoparticles are far brighter than organic dyes and can only be lit by a single light source. Thus, compared to utilising organic dyes, using fluorescent QDs can produce images with noticeably higher contrast for less money. A disadvantage of QDs is that they are made of potentially harmful materials.

1.6.5 SENSING

The lab-on-a-chip approach is comparable to nanotechnology for sensing. Using sensor test chips with hundreds of nanowires that can recognise proteins and other signs left behind by cancer cells, a patient's blood could be utilised to detect and diagnose cancer in its early stages[15].

1.6.6 FOOD

The main uses of nanotechnology in the food business are in the development of sensors, food contact materials, and encapsulation and emulsions. Garber refers to the cultivation, manufacture, packaging, and processing of food using nanoparticles as "nanofood." The FSAI list of uses for nanofood includes sensory improvements (flavour, colour, and texture modification), increased absorption, targeted delivery of nutrition bioactive compounds, stabilisation of active ingredients like nutraceuticals in food sources, packaging and product improvement to extend shelf life, sensors for food safety, and antimicrobials to eradicate pathogenic microbes in food47,48. Bionanocomposites are hybrid nanoparticles with enhanced mechanical, thermal, and gas characteristics. They are used in packaging to assist increase food's shelf life. As a result, packaging will rely less on plastic, which is safe for the environment. Zein, a prolamin and a major component of maize protein, is used as an example. To develop a biodegradable zein film with improved tensile and water barrier qualities, zein can be dissolved in ethanol or acetone49. In Australia, white bread is supplemented with omega-3 fatty acids using nanocapsules. Non-toxic nanoscale insecticides are being developed in Asia to interact with the seed coverings of weed seeds and prevent germination50. Because of this, the use of nanotechnology to food is rapidly growing and affects every link in the food chain[16].

1.7 COPPER OXIDE NANOPARTICLES

Copper oxide nanoparticles are brownish-black in hue when they are in powder form. They can become metallic copper when heated to a high temperature and exposed to hydrogen or carbon monoxide. They are categorised as harmful to aquatic life, harmful to the environment, and harmful to humans.

1.7.1 CHEMICAL PROPERTIES

The table below lists the chemical characteristics of copper oxide nanoparticles.

CHEMICAL SYMBOL	CuO
CAS No	1317-38-0
GROUP	Copper-11 Oxygen-16
ELECTRONIC CONFIGURATION	Copper-[Ar]$3d^{10}4S^{1}$ Oxygen-[He]$2S^{2}2P^{4}$

Table 1.1 Chemical data of CuO nanoparticles

ELEMENT	**CONTENT (%)**

Copper	79.87
Oxygen	20.10

Table 1.2 Chemical composition of CuO nanoparticles

1.7.2 PHYSICAL PROPERTIES

The physical properties of copper oxide nanoparticles are given in the following table.

PROPERTIES	**METRIC**	**IMPERIAL**
Density	6.31 g/cm^3	0.227lb/in^3
Molar mass	79.55 g/mol	

Table 1.3 Physical properties of CuO nanoparticles

1.7.3 THERMAL PROPERTIES

The thermal properties of copper oxide nanoparticles are provided in the table below.

PROPERTIES	METRIC	IMPERIAL
Melting point	1201^0C	2194^0F
Boiling point	2000^0C	3632^0F

Table 1.4 Thermal properties of CuO nanoparticles

1.7.4 MANUFACTURING PROCESS

Copper oxide nanoparticles can be produced through aqueous precipitation. Copper acetate serves as a precursor while sodium hydroxide acts as a stabilising agent in this process. The single phase monoclinic structure of the copper oxide nanoparticles is highlighted by X-ray diffraction. The rectangular form of the copper oxide nanoparticles is revealed via scanning electron microscopy.

1.7.5 APPLICATIONS

The primary applications for copper oxide nanoparticles are as follows:
As a catalyst to speed up combustion in rocket propellant; as magnetic storage media; as gas sensors; as near-infrared tilters; as photoconductive and photothermal applications; as semiconductors; as solar energy conversion; and as high-tech superconductors[17]. It can reduce pressure index, significantly increase homogenous propellant burning rate, and work better as a catalyst for AP composite propellants.

1.8 NANOCURCUMIN

1.8.1 WHAT IS NANOCURCUMIN?

Curcumin particles have been reduced to nano-sized (insignificant) particles, known as nanocurcumin, to enhance transport, solubility, and absorption in the body. Turmeric's main dynamic fixer is curcumin. Curcuma longa, a rhizomatous herbaceous perennial plant in the ginger family (Zingiberaceae), produces turmeric, also referred to as the golden curry spice. It has been used in Ayurvedic and Chinese medicine for hundreds of years to cure a variety of diseases.

1.8.2 CHEMICAL COMPOSITION OF NANOCURCUMIN

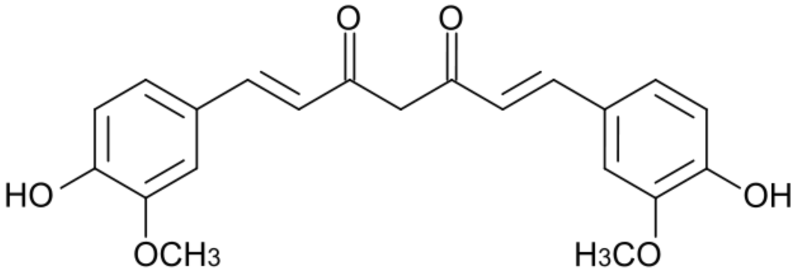


Figure 1.4 Chemical structure of nanocurcumin

Nano curcumin comprises minerals (3.5%), carbohydrates (96.4%), moisture (13.1%), protein (6.3%), and fat (5.1%) according to chemical analysis. Its extracts produce the curcuminoids curcumin (77%), demethoxycurcumin (DMC) (17%), and bisdemethoxycurcumin (BDMC). Curcuminoids, particularly curcumin, are used in supplements and pharmaceuticals [8]. Another name for the symmetric chemical curcumin is diferuloylmethane. Its molecular weight is 368.38 and its chemical formula is C21H20O6. Two aromatic ring systems with o-methoxy phenolic groups are joined by a seven-carbon linker made up of an, -unsaturated -diketone moiety to form the structure's three chemical parts.

1.8.3 CLINICAL EVIDENCE

- **ANTI-INFLAMMATORY**

  Sixty COVID-19 patients were randomly assigned to the control and nano-curcumin groups, and the nano-curcumin dose was 240 mg/day for seven days.The laboratory values in the patients were recorded on days 0 and 7. Between the control and nano-curcumin groups, there were statistically significant differences in the serum levels of IL-1 (p = 0.042). According to the results, nano-curcumin may be utilised in addition to traditional treatment to act as an anti-inflammatory and stop the effects of inflammation.

- **ANTISEPSIS**

  The goal of the current trial was to investigate the effects of dietary addition of nano-curcumin on haematological markers in critically ill sepsis patients. Nano-curcumin or a placebo was given to 14 ICU patients at random for 10 days. Interleukin-8 levels in the nano-curcumin group were significantly lower than those in the placebo group after 10 days of treatment. At the conclusion of the research, the total lymphocyte count was considerably higher in the nano-curcumin group than in the placebo group (p =.04). For patients with severe sepsis, nano-curcumin may be a useful adjuvant therapy when used in combination.

1.8.4 BIOAVAILABILTY OF CURCUMIN

Curcumin nanoparticles increase the solubility of curcumin by boosting bioavailability of curcumin by more than five times when compared to pure curcumin powder.In this study, a high-pressure homogenization approach is used to make curcumin more soluble, and various stabilisers produce their effects by working through various stabilisation pathways. For producing curcumin nanocrystals, the high pressure homogenization (HPH) technique is preferable to other methods. This formulation improves the stability, saturation solubility, and dissolution rate of nanocrystal-containing medicine capsules. Numerous testing have revealed that, compared to commercial items, the dissolving behaviour has significantly improved.Poorly solubility-behaving medications can have their bioavailability enhanced by improving the solubility behaviour in drug-loaded solid dosage forms built of nanocrystals.

HEALTH BENEFITS

Nanocurcumin supplements help the immune system and prevent bacterial infection. Processing occurs to produce anti-inflammatory properties. Both blood pressure and cholesterol levels are reduced.Control your period, unwind and calm your thoughts, and encourage deeper slumber.strengthens bones while lowering joint pain and improving lung health.maintain normal insulin levels and keep blood sugar under control.

SAFETY

Fatalities related to the medication or significant adverse effects were discovered, which strengthens the immune system and prevents bacterial infection. No patients dropped out of the research as a result of unfavourable drug effects. Nanocurcumin is safe and may raise survival rates in ALS patients, particularly those who already have bulbar symptoms**.**

1.8.5 MECHANISM OF ACTION:

By boosting the immune system and preventing bacterial infection Nanocurcumin particles swiftly dissolve in water by up to 98% more fast than curcumin does, increasing the body's maximal absorption capacity. The solubility of nanocurcumin, which has a size range of 30 nm to 100 nm, is hundreds of times more than that of conventional curcumin. The curcumin nanoparticles' extremely small size helps them to penetrate quickly, boosting their therapeutic activity in most of the body's organs[18].

1.9 CHARACTERISATION TECHNIQUES

1.9.1 SCANNING ELECTRON MICROSCOPY[SEM]

By boosting the immune system and preventing bacterial infection In a scanning electron microscope (SEM), a focused electron beam is used to scan a sample's surface and produce images of the sample. The signals generated by the interactions of the electrons with the sample atoms show the surface topography and chemical composition of the sample. By combining the electron beam's position and the strength of the signal being detected as it is being scanned in a raster scan pattern, an image is produced.The most common SEM mode makes use of an Everhart-Thornley detector to discover secondary electrons generated by excited atoms. By boosting the immune system and preventing bacterial infection One element that impacts the quantity of secondary electrons that may be detected and, as a result, the signal strength, is the topography of the material. Some SEMs have resolutions greater than 1 nm.Specimens are inspected using specialised equipment in a variety of cryogenic or high temperatures, high vacuum in a standard SEM, or low vacuum or wet conditions in a variable pressure or environmental SEM[19]. By boosting the immune system and preventing bacterial infection To outperform the transmission electron microscope, Mafred von Ardenne developed the first scanning electron microscope in 1937. He used high-resolution power to scan a small raster after focusing an electron beam on it. He also made an effort to reduce chromatic aberrations in images taken using transmission electron microscopes. In 1965, Cambridge Scientific Instrument Company manufactured a fully functional scanning electron microscope under the name Stereoscan. Scientists and research organisations continued their investigations.

BASIC PRINCIPLE

Immune defences are boosted, and bacterial infection is avoided thanks to The accelerated electrons in a SEM release significant quantities of kinetic energy as a variety of signals that are produced by electron-sample interactions as incident electrons are decelerated in the solid sample. These signals include heat, photons (distinct X-rays used for elemental analysis and continuum X-rays), cathodoluminescence-CL, secondary electrons (which produce SEM images), backscattered electrons (BSE), diffracted backscattered electrons (EBSD), and backscattered electrons (BSE). The use of secondary electrons and backscattered electrons for imaging samples is common. In contrast to secondary electrons, which are best used to show morphology and topography on materials, backscattered electrons are best used to illustrate contrasts in composition in multiphase samples (i.e., for rapid phase discrimination). X-rays are released as a result of inelastic collisions between incident electrons and electrons in particular atomic orbitals (shells) in the sample. When excited electrons in an element's different shells return to lower energy states, the difference in their energy levels produces X-rays with a constant wavelength. Each component of a mineral that is "excited" by the electron beam as a result generates unique X-rays. SEM testing is classified as "non-destructive" since no sample volume is lost to the x-rays created by the interactions of the electrons, allowing for repeated analysis of the same materials[21].

1.9.2 TRANSMISSION ELECTRON MICROSCOPY[TEM]

Transmission electron microscopy (TEM) is a type of microscopy in which an electron beam is sent through a material to produce a picture. The specimen is typically a suspension on a grid or an ultrathin slice that is no thicker than 100 nm. The interactions of the electrons with the specimen as the beam travels through it result in an image. The picture is then magnified and focused using an imaging device, such as a fluorescent screen, a piece of photographic film, or a sensor similar to a scintillator connected to a charge-coupled device. Transmission electron microscopes have a far higher resolution than light microscopes because of the short de Broglie wavelength of electrons. This enables the equipment to record incredibly fine detail since a single column of atoms is hundreds of times smaller than a resolvable object viewed in a light microscope. Transmission electron microscopy is a vital analytical method in the physical, chemical, and biological sciences. TEMs are used in the study of cancer, virology, materials science, as well as other fields including palaeontology and palynology, in addition to pollution, nanotechnology, and semiconductor research[22].

BASIC PRINCIPLE

At the top of the microscope, an electron source emits electrons that travel through a vacuum in the instrument's column. Electromagnetic lenses are utilised to focus the electrons, resulting in a very small beam that is subsequently directed through the target item. The electrons then have an impact on the detector as they pass through the object. The compositional density and crystal orientation of the sample impact how incident electrons scatter and disappear from the beam in conventional bright field imaging.

A "shadow image" of the specimen is produced by the amount of unscattered electrons, with different densities of the specimen's constituent parts depicted in diverse shades of black. It is also possible to turn the specimen while taking a number of pictures, creating a 3D representation of the specimen.

Additionally, samples with regular atomic structure (crystalline material) can be used to study the crystal structure using electron diffraction. Discrete zones of electron localization generated by positive interference in the back focal plane may then be observed by mapping the back focal plane to the imaging device. The diffraction patterns can then be used to identify the specimen's crystal structure. The primary electron beam interacting with the material results in X-ray emission, which can be detected by an energy-dispersive spectrometer (EDS) inside the TEM. Because the resulting X-ray energy is a good indicator of the atomic structure of the element they came from, the spectra generated can be used to identify the constituent elements.

It is also possible to detect the energy lost due to the inelastic scattering of electrons in specimen transmission (EELS). These results allow for the inference of the elemental composition, chemical bonds, and electrical properties of the valence and conduction bands[23].

HRTEM

High-resolution TEM uses a somewhat different technology than other TEM types, resulting in a high resolution that can structurally describe samples at the atomic level. It is therefore a very useful tool for studying any nanoscale structures.

Due to the fact that objects at this scale are frequently extremely difficult to view and define, using this method offers up a brand-new field of study for researchers working in subjects like nanotechnology, nanomedicine, or material science.

It is essential to comprehend electron scattering in order to comprehend how HR-TEM works. Transmitted electrons interact with the sample's atoms through elastic and inelastic scattering. Inelastically scattered electrons undergo an energy shift after passing through the sample, in contrast to elastically scattered electrons, which maintain their initial transmitted energy and are consequently more valuable for the interpretation of the data.

When the elastically scattered electrons leave the sample and pass by the microscope's lenses, a high-resolution image is produced. Electron energy loss spectroscopy, or EELS, can be utilised to retrieve data from the material even though this method often does not use inelastically scattered electrons. The data produced by the scattering of photons and electrons can be used in analytical techniques like Raman spectroscopy and medical ultrasonography. Since particle scattering differs depending on the molecular or atomic structure of the sample, it provides important information[24].

1.9.3 POWDER X-RAY DIFFRACTION [PXRD] STUDY

X-ray powder diffraction (XRD), a fast analytical technique, is frequently employed to ascertain the phase of crystalline materials. Additionally, it can disclose details about unit cell dimensions. The substance being studied has been finely ground, homogenised, and the bulk composition has been estimated on average.

BASIC PRINCIPLE

Monochromatic X-rays and a crystalline sample interact positively. X-ray diffraction is based on constructive interference of monochromatic X-rays with a crystalline material. The X-rays are generated by a cathode ray tube, filtered to provide monochromatic radiation, collimated, and directed at the sample. The incident rays interact with the sample to produce constructive interference (and a diffracted ray) when the conditions are in compliance with Bragg's Law (n=2d sin). This law establishes a relationship between the wavelength of electromagnetic radiation and the lattice spacing and diffraction angle in a crystalline sample. These diffracted X-rays are then recognised, investigated, and counted. The sample should be scanned through a range of 2 angles in order to capture all feasible lattice diffraction directions due to the random orientation of the powdered material. The conversion of the diffraction peaks to d-spacings allows for mineral identification because each mineral has a unique set of d-spacings. This is frequently done by making comparisons between the d-spacings and recognised reference patterns. All diffraction methods are based on the generation of X-rays in an X-ray tube. These X-rays are directed at the sample, and the diffracted rays are recorded. All types of diffraction depend heavily on the angle between incident and diffracted rays. Beyond this, the equipment used for single crystal and powder diffraction varies[25].

1.9.4 INFRA RED SPECTROSCOPY

One of the most well-known and often used spectroscopic techniques, it is mostly employed by inorganic and organic chemists because to its usefulness in detecting the structures of compounds and identifying them.To construct an infrared spectrum, infrared spectroscopy is carried out using an infrared spectrometer, also referred to as a spectrophotometer[26].

BASIC PRINCIPLE

Infrared spectroscopy is the study of how infrared light interacts with molecules. With an infrared wavelength between 2,500 and 16,000 nm and a frequency range between 4000 $cm^{-1}$to 600 cm^{-1} with Hz as its fundamental frequency, this infrared spectrum is best suited for analysing organic compounds. Although the photon energy in this area of the infrared spectrum (between 1 and 15 kcal/mole) is insufficient to excite electrons, it may do so for atoms and groups that are covalently bound. It is well known that, in addition to the simple rotation of groups around a single bond, molecules experience a range of vibrational motions that are specific to their atoms. Then, infrared light, which has an energy akin to vibrations, is absorbed by all biological materials. Infrared spectrometers allow chemists to acquire absorption spectra for compounds that solely reflect the molecular structures which is conceptually similar to other spectrometers[27].

1.9.5 ULTRA VIOLET- VISIBLE SPECTROSCOPY

The field of spectroscopy known as UV-Vis spectroscopy, also known as ultraviolet-visible spectroscopy, focuses on how efficiently a sample absorbs UV and visible light. It is a frequently used technique in analytical chemistry, biochemistry, and materials research. UV-Vis spectroscopy can be used to determine a molecule's concentration in a solution, its functional group composition, and its electrical and geometric structure.

The main objective of UV spectroscopy is to determine a sample's transmission or absorption characteristics in the ultraviolet (UV) region of the electromagnetic spectrum. The amount of light absorbed by a sample at different wavelengths can be used to gather details about a sample's composition, electrical structure, and other properties. There are many uses for UV spectroscopy, including estimating and measuring the concentration of a certain ingredient in a combination describing the purity of the sample.

SOLVENTS USED

- Depending on the application and the type of sample being studied, a different UV spectroscopy solvent is utilised. Some typical UV spectroscopy solvents are listed below:

- Alcohols: For UV spectroscopy of organic molecules, alcohols like methanol and ethanol are widely used as solvents.Water: Polar compounds, such as acids, bases, and biomolecules, are widely studied using UV spectroscopy in which water is used as a solvent.

- Acetonitrile: Acetonitrile is a polar, aprotic solvent that is often used for UV spectroscopy of polar compounds and for samples that are not soluble in water or alcohols.

- Dichloromethane: Dichloromethane (DCM) is a non-polar solvent that is often used for UV spectroscopy of non-polar compounds.

BASIC PRINCIPLE

Spectroscopy is fundamentally concerned with the interactions between light and matter.

When light is absorbed by matter, there is an increase in atomic or molecular energy. Electrons are stimulated from their ground state to a higher energy state when UV radiation is absorbed. Molecules with -electrons or non-bonding electrons (n-electrons) can absorb the energy of ultraviolet light to excite their electrons to higher anti-bonding molecular orbitals. Longer light beams can be absorbed when electrons are stimulated more readily. It is possible for there to be four separate transition types (π–π, *n–π*, σ–σ, *and n–σ*), which can be arranged as follows: σ–σ* > n–σ* > π–π* > n–π*. A spectrum is produced when a chemical compound absorbs UV light, and this spectrum can be used to identify the substance.

Using ultraviolet and visible spectroscopy, also known as electronic spectroscopy, one may calculate the amount of double bond conjugation and aromatic conjugation present in a molecule. Since HOMO refers for Highest Occupied Molecular Orbital and LUMO is for Lowest Unoccupied Molecular Orbital, electrons must switch from their HOMO to LUMO states as a result. When conjugation increases, the HOMO-LUMO gap gets smaller. [28].

1.9.6 BRUNAUER-EMMETT-TELLER [BET] ANALYSIS

The type II isotherm seen in non-porous materials can be utilised to determine An inert gas, commonly nitrogen, is adsorbed on the surface of a solid substance in physical gas adsorption. This happens on the interior and exterior surfaces because porous materials have pores. BET analysis, which evaluates the BET surface area by gas adsorption, is the most well-known technique. At 77 K, the temperature of liquid nitrogen, when nitrogen is absorbed, an isotherm known as adsorption, or BET isotherm, is produced. You can measure this isotherm over porous and non-porous materials. The BET surface area. Porous materials frequently provide either a type I or type IV isotherm depending on the type of pores present. BET study over type I isotherm materials requires a particular technique in order to accurately determine the BET surface area. In some cases, such as for zeolites or activated carbons, the use of argon or carbon dioxide adsorption is typically preferred over nitrogen adsorption to precisely probe the tiny micropores. Low BET surface area samples can be efficiently characterised by a BET research utilising krypton gas adsorption. In spite of the fact that only a tiny section of the isotherm can be accurately measured in these conditions, the BET surface area can be calculated.

BASIC PRINCIPLE

The theory of gas molecule monolayer formation on a solid surface is used to compute the specific surface area. The BET model, which uses a linearized version of the BET equation, transforms the experimental adsorption isotherm into a BET plot, allowing the monolayer volume to be computed. This BET analysis then uses the cross section area of the gas molecule and the monolayer volume to calculate the overall specific surface area expressed in m2/g. Gas adsorption can also be used to identify the presence of pores in terms of pore volume and pore size distribution in addition to BET analysis. The pore size distribution can be determined using the capillary condensation in pores principle because capillary condensation in pores is pore size dependant. The pressure needs to be higher in larger holes in order to produce condensation. Smaller holes are filled in a similar manner at lower adsorbing gas partial pressures. Before the measurement, the sample is pre-treated at a high temperature in a vacuum or a flowing gas to remove any contaminants. Given that too low or too high a temperature could drastically modify the BET surface area acquired from the subsequent BET study, pre-treatment settings must be matched to the characteristics of the materials.

The following adsorption analysis can be distinguished between two general techniques:

Q surf M3 analyzer can be used to make the flow measurements, and the report only includes a single number for the total pore volume or BET area. Quantachrome Autosorb-6B (N2 or CO2), Micromeritics TriStar 3000 (N2), Micromeritics Gemini, or Micromeritics ASAP 2020 (Ar or Kr) are some examples of the instruments that can be used to perform volumetric adsorption measurements. The report can range from a single value for the BET surface area to a full report on isotherm, specific BET surface area, total pore volume, and pore size distribution, going beyond a simple bet analysis [29].

1.9.8 ANTIBACTERIAL STUDY

Only chemicals having antibacterial and antiviral capabilities are able to effectively kill or stop the spread of bacteria and viruses without seriously damaging the tissues nearby[30].

Disk Diffusion Method

Because the test plant extract diffuses across the agar media planted with the test microorganism from its reservoir, the disc diffusion technique (DDM) is classified as a method (ADM). On top of an agar surface, a filter paper disc is frequently used as the reservoir. If the isolated compounds or plant extracts under study are microbiologically active, an inhibitory zone emerges around the filter paper disc during incubation. The diameter of the inhibition zone is the best indicator of the potency of plant extracts or particular compounds against microorganisms. The water-insoluble EO and its components do not sufficiently diffuse from a filter paper disc into the agar medium, making DDM an inappropriate method for lipophilic extracts (such as EOs). Because of this, EOs give DDM negative results or inhibitory zones that are smaller than with other tests, including the BDM[31].

Well Diffusion Method

Dilution testing is used to determine the the minimum concentration of an antimicrobial agent (in mg/ml) required to prevent the growth of the bacteria. Two-fold dilutions of the antimicrobial agent are added directly to an agar pour, a broth tube, or a micro-broth panel to achieve this.

Antimicrobial studies for plant extracts and secondary metabolites are conducted using agar well diffusion to evaluate the extracts' capacity to inhibit bacterial growth. The agar well diffusion method is commonly employed to assess the antibacterial activity of plant or microorganism extracts.Similar to the disk-diffusion method, the agar plate surface is inoculated by spreading a volume of the microbial inoculum over the entire surface. Antimicrobial activity was determined by measuring the zone of inhibition, which included the wells' diameter, after the incubation period[32].

1.10 OBJECTIVES

- To synthesize curcumin and CuO nanoparticles by green synthesis methods.

- To functionalize the synthesized CuO nanoparticles with nanocurcumin.

- Characterize the synthesized nanoparticles using UV-visible absorption spectroscopy, IR spectroscopy, Scanning Electron Microscopy, Transmission Electron Microscopy, BET studies, X-ray Photo Electron Spectroscopy and Powder XRD analysis.

- Study and compare the dye absorption ability of the synthesized samples using methylene blue, methyl orange and Eriochrome black-T as model dyes.

- Examine the antibacterial efficiency of the synthesized sample against E.Coli.

Chapter 2

Review of Literature

In the modern world, the subject of water analysis has grown significantly. Heavy metals from human activities or industrial processes, such as organic and inorganic solutes (Hg, As, Pb, Cr, Cu, Zn, and others), are the principal causes of water contamination.Different human health issues are caused by this water contamination, along with the extinction of biodiversity.The level of water contamination, which is rising daily, calls for better technological advancements. Nanotechnology offers several benefits for enhancing environmental technologies and developing new, more effective methods for reducing water pollution. Here this chapter provides a summary of recent developments in nanotechnologies for water and wastewater treatment processes, including nanobased materials like copper oxide nanoparticles (CuO NPs). As a result of their small size, high surface area, natural abundance of starting material for synthesis, and low-cost manufacturing processes, copper oxide nanoparticles (CuO NPs) are frequently studied as efficient adsorbent materials. This study also assessed the effectiveness of CuO NPs functionalized with nanocurcumin for the adsorption of contaminants. By analysing a variety of literature, we found that CuO NPs can be used as an adsorbent for the mitigation of various contaminants from the aqueous environment.

Rupesh Kumar Basniwal and co-workers(2011),reported the preparation of nanoparticles of curcumin with a view to improve its aqueous-phase solubility and examine the effect on its antimicrobial properties. Wet-milling was used to prepare curcumin nanoparticles, (nanocurcumin) which were discovered to have a specific particle size range between 2 and 40 nm. In contrast to curcumin, it was found that nanocurcumin was freely dispersible in water without the use of any surfactants. During the synthesis of the nanoparticles, the chemical structure of nanocurcumin remained unchanged from that of curcumin. For a number of bacterial and fungal species, a minimum inhibitory concentration of nanocurcumin was established and compared to that of curcumin. It was found that curcumin was significantly less efficient than the aqueous dispersion of nanocurcumin against Staphylococcus aureus, Bacillus subtilis, Escherichia coli, Pseudomonas aeruginosa, Penicillium notatum, and Aspergillus niger. The outcomes showed that curcumin's water solubility and antibacterial activity were significantly enhanced by particle size reduction up to the nano range. The activity of nanocurcumin was stronger against Gram-positive bacteria than Gram-negative bacteria for the chosen pathogens. Additionally, its antibacterial activity was far more effective than its antifungal activity. The transmission electron microscopy (TEM) analysis used to investigate the mechanism of curcumin nanoparticles' antibacterial action showed that these particles entered the bacterial cell by fully rupturing the cell wall, which caused cell death.[32]

Judy Gopal et al.(2015) from Department of Molecular Biotechnology, Konkuk University,Korea employed two different sonication methods, water bath type and probe sonication,to develop this one-step, water-based technique for extracting curcumin directly from turmeric.They used two separate sonication methods, water bath type and probe sonication.55% of the curcumin was extracted from the water using the probe sonication technique with a 20 kHz frequency and a 5 minute sonication period.This yield is even greater than that attained by ethanol-based solvent-based extraction techniques.The physical change of micro-curcumin to nano-curcumin by ultrasonic means is demonstrated to be the cause of curcumin's improved water solubility and subsequent efficient extraction.The findings of this study recommend using probe ultrasonication to extract curcumin from turmeric using water in a single step. Due to curcumin's insolubility in water, this study also offers a solution for the bioavailability issue. Curcumin was nanosized utilising ultrasonication techniques. In this communication, the outcomes and verification of these findings are given.[33]

Copper sulphate 5-hydrate was used as a precursor and NaOH as a stabilising agent in the aqueous precipitation process used by Ghulam Mustafa et al. (2013) to create cupric oxide (CuO) nanoparticles (NPs). This results in the formation of CuO-NPs on a huge scale, which are then used to remove the dye methylene blue (MB). The CuO NPs were identified in order to examine their composition and structure using XRD, which indicates the monoclinic single phase structure. Scanning electron microscopy (SEM) was used to examine the surface morphology of these NPs. Additionally, the impact of optimisation factors including temperature, concentration, and time was also looked at. A spectrophotometric approach was employed to assess how well NPs removed MB from an aqueous solution.At temperatures ranging from 303 to 318 K + 2K, the equilibrium adsorption of the cationic dye (MB) was conducted. The Langmuir, Freundlich, and Dubinin-Radushkevich adsorption isotherm equations were used, and the graphical technique was used to get the values of each constant. To assess the viability of the method, thermodynamic and kinetic experiments were also carried out. The highest MB elimination recorded was 88.93%. Additionally, using the pH drift method, the pH of the adsorbent's point zero charge (pHPZC) was calculated. According to the findings, the aqueous precipitation approach offers a dependable and affordable way to create CuO-NPs that may be utilised to successfully remove colours from effluents. For the cleanup of industrial waste, this technology is advantageous.[34]

Aloe vera gel, which has a wide range of medical applications due to its anti-inflammatory, antibacterial, anticancer, and wound healing capabilities, was utilised by S Rajeshkumar et al. (2019) to synthesis copper oxide nanoparticles. After that, they were characterized using FT-IR and UV-visible spectrophotometry. It was also investigated to see if oral pathogens such Staphylococcus aureus, Streptococcus mutans, Enterococcus faecalis, and Pseudomonas sp. had any anticaricinogenic properties. An intense surface plasmon resonance between 250 and 300 was visible in the synthesised nanoparticles' UV absorption spectra. By using FT-IR, the functional groups in the copper oxide nanoparticles were identified, and it was also demonstrated that these groups had strong anticariogenic activity. Additionally, copper oxide nanoparticles effectively fight off a variety of diseases as well as germs that are resistant to antibiotics. They reported Copper oxide nanoparticles synthesized using Aloe vera gel shown to have an excellent anticarcinogenic activity and has a wide array of dental applications[35]

In 2015, R Subbiya and M Masilamani Selvam reported on the benefits of green nanoparticle synthesis. The surface plasmon resonance phenomenon caused the synthesised copper oxide nanoparticles to turn dark brown, which served as proof. Transmission electron microscopy (TEM), Fourier transmission infrared spectroscopy (FT-IR), and ultraviolet-visible spectroscopy were used for the characterization examinations. From FRAP and hydrogen peroxide scavenging assays, leaf extract containing copper oxide nanoparticles shown strong anti-oxidant efficacy. The study showed that copperoxide nanoparticles made biologically were an efficient lung cancer treatment[36].

Curcumin is typically encapsulated in nanocarriers for targeted administration, according to Sandhuli S and his coworkers' paper from the year 2021. The disadvantages of curcumin, such as its limited absorption, rapid metabolism, and low water solubility, are eliminated by this approach. In their research, they created nanoparticles of curcumin without employing nanocarriers because worries about the degradation of nanocarriers' products had been raised. Curcumin exhibits distinct and highly crystallin peaks in X-ray diffraction, showing its identification.
The presence of all of curcumin's functional groups in nanocurcumin was confirmed by Fourier Transform infrared spectroscopy. The completely spherical form of nanocurcumin was visible in TEM and SEM pictures. Nanocurcumin formulations can effortlessly disperse in water even if cucurmin is not water soluble[37].

According to Srividhya Parthasarathy and colleagues (2020), researchers are growing more interested in the topic of nanotechnology as a safe way to create nanoparticles. The creation of copper nanoparticles was aided in this experiment by the use of Plectranthus amboinicus, also referred to as Mexican mint and identified as Coleus amboinicus leaf extract. We report a green synthesis that makes stable copper nanoparticles quickly and easily from Plectranthus amboinicus leaf extract.They report a green synthesis that makes stable copper nanoparticles quickly and easily from Plectranthus amboinicus leaf extract. To make the copper nanoparticles, the naturally occurring copper sulphate solution was employed as a precursor. The plant extract from Plectranthus amboinicus showed excellent reducing and stabilising properties. With the help of X-Ray Diffraction (XRD), Zeta Potential, and ultraviolet-visible spectroscopy, it was determined that copper nanoparticles had been produced. The UV-Spectrometer analysis displays a characteristic peak that may indicate the formation of copper nanoparticles. Particle sizes between 16 and 25 nm were seen in the XRD pattern, which suggests that the copper nanoparticles had high crystallinity. Zeta potential was used to determine the nanoparticles' charge and size distribution, which showed significant stability. This method exhibits efficiency, usability, and pollution-free operation[38].

Mayada Qasem reported that Copper oxide (CuO) nanoparticles (NPs) and CuO nanograins were created using a revolutionary easy green production method.(NGs) by employing curcumin (a natural and non-toxic dietary spice) as the copper salt precursor and cupric chloride dihydrated as at extremely basic conditions, a conjugated agent acting with or without cetyl trimethylammonium bromide stabilises the system. Studying the effects of a number of parameters, starting with the copper salt precursor, curcumin content, pH, and temperature, allowed for the optimisation of the synthesis of these curcumin linked CuO nanoparticles.The produced

CuO NPs/NGs were characterised using scanning electron microscopy linked to an energy dispersive X-ray analyzer, thermogravimetric analysis, X-ray diffraction, UV-visible spectroscopy, fluorescence spectroscopy, and zeta potential. In this study, copper oxide nanograins that had been synthesised were discovered to be effective nano-catalysts for the reduction of methylene blue. It was also discovered that nanograins had better catalytic activity than spherical shape nanoparticles, highlighting the importance of the shape of the CuO particles for their nanocatalytic activity[39].

Curcumin has been used in numerous anti-microbial research because of its low side effects and extensive traditional applications. Despite having a wide range of effects, the intrinsic physicochemical characteristics such as low bioavailability, poor water solubility, photodegradation, chemical instability, short half-life and fast metabolism of curcumin derivatives limit their pharmaceutical importance. To overcome these drawbacks and improve the therapeutic ability of curcuminoids, novel approaches have been attempted by Simin Sharifi et al.(2020) recently. Nanoparticulate drug delivery systems can increase the efficiency of curcumin in several diseases, especially infectious diseases. These innovative strategies include polymeric nanoparticles, hydrogels, nanoemulsion, nanocomposite, nanofibers, liposome, nanostructured lipid carriers (NLCs), polymeric micelles, quantum dots, polymeric blend films and nanomaterial-based combination of curcumin with other anti-bacterial agents. Integration of curcumin in these delivery systems has displayed to improve their solubility, bioavailability, transmembrane permeability, prolong plasma half-life, long-term stability, target-specific delivery and upgraded the therapeutic effects. In this review paper, a range of in vitro and in vivo studies have been critically discussed to explore the therapeutic viability and pharmaceutical significance of the nano-formulated delivery systems to elevate the anti-bacterial activities of curcumin and its derivatives. Curcumin has been shown to enhance the therapeutic effects of various delivery methods by increasing their solubility, bioavailability, transmembrane permeability, plasma half-life, long-term stability, and target-specific delivery. In order to investigate the therapeutic viability and pharmacological importance of the nano-formulated delivery systems to enhance the anti-bacterial activities of curcumin and its derivatives, a variety of in vitro and in vivo research have been critically reviewed [40].

Joshua O. Ighalo et al (2021) studied adsorption of pollutants by CuO nanoparticles.CuO nanoparticles have been used to study the adsorption of contaminants, according to Joshua O. Ighalo et al.Their goals were to synthesise existing research findings, identify knowledge gaps, and predict potential research fields. The most widely used methods for the synthesis of CuO NPs use conventional chemical procedures like precipitation and microwave heating. The greatest reported adsorption capacity for CuO NPs was 3152 mg/g for fluoride, demonstrating that it can remove more than three times its weight in fluoride from the aqueous phase. The pseudo-second order model (R2 > 0.99) and Langmuir model (R2 > 0.99) are the best-fit kinetics and isotherm models for adsorption utilising CuO NPs, respectively, for dyes and heavy metals. Analysis of the thermodynamics showed that the process of adsorption by CuO NPs was primarily endothermic and spontaneous CuO NPs can be recycled up to five times with an average >80% recovery of contaminants. Future research should focus on mechanistic statistical physics studies, competitive adsorption, and column experiments. CuO NPs use has a detrimental ecotoxicological effect, hence extra care must be made to remove/recover it from wastewater before releasing it into the environment. CuO NPs are believed to be a useful adsorbent for the reduction of different contaminants from the aquatic environment.[41]

Mostafa M. Mohamed and co-workers developed a simple, achievable, and reproducible method for preparation of biocompatible nanocurcumin. To synthesize curcumin nanoparticles for their investigation they used the solvent anti-solvent precipitation approach.They tested various organic solvents for biocompatibility in order to determine w the best option for safety and curcumin solubility. Additionally, they investigated how adding stabilisers might affect the nanocurcumin's production and stability. The stabilised nanosample was then examined for luminescence and water solubility.They then noticed that nanocurcumin dissolved in acetone produced clear, amber-yellow nanosuspension that was satisfactory in concentration and free of precipitate. The nanosample was monodispersed by minimising the stirring rate and time. The maximum solubility and luminescence were also found in stabilised nanocurcumin. The rapid fluorescent feature of the soluble nanosized curcumin, they deduced, would make this herbal nano-candidate suitable for use as a theranostic agent.[42].

Based on the literature review mentioned above we used the green synthesis approach to synthesised copperoxide nanoparticles using aloe-vera extract and copper sulphate as precursors. We also synthesised Nanocurcumin Using the ultrasonication process which is again a greener approach. After incorporating nanocurcumin to the synthesised copper oxide nanoparticles, we carried out their dye adsorption studies and anti-bacterial efficiency.

Chapter 3

Materials and Methods

3.1 EXPERIMENTAL SECTION

Copper (II) sulphate penta hydrate ($CuSO_4.5H_2O$), Sodium Hydroxide (NaOH) Pellets, Acetone, Eriochrome Black T, Methyl orange and Methylene blue were purchased from NICE chemicals Pvt. Limited. Aloe-vera leaves and rhizomes of turmeric were collected from the nearby Garden. All chemicals were used as received without further purification. Double distilled water was used throughout the experiment.

3.2 SYNTHESIS OF NANO-CURCUMIN

Turmeric rhizomes were cleaned well;the fingers and the mother rhizomes were separated and blanched in a closed pot filled with ¾ of water for 30 minutes. The rhizomes were dried under sunlight until moisture content was reduced to 10%.Then it was ground using grinder to obtain turmeric powder. For the extraction experiment, 0.5 g of turmeric powder was dissolved in 10 ml acetone taken in conical flask followed by filtration through whatman filter paper[grade:40]. It is then ultrasonicated in an ultrasonic bath with tank capacity 0.8l. During the experiment, the Erlenmeyer containing the sample was covered with parafilm inorder to prevent solvent loss. The sonication was performed for 40 min within the temperature range 25-40°C.The resulting solution contains nano curcumin and was stored in an airtight sample bottle. The synthesis route is outlined in fig.3.1.

CURCUMIN RHIZOMES

CURCUMIN RHIZOMES SUN DRIED

0.5g TURMERIC WEIGHED

0.5g TURMERIC POWDER DISSOLVED IN 10 ml ACETONE

FILTRATION THROUGH WHATMANN 4O FILTERPAPER

ULTRASONICATED FOR 40 MINS AT 25-40^0 C

OBTAINED NANOCURCUMIN SOLUTION IN ACETONE

Fig 3.1 Synthesis route of Nanocurcumin

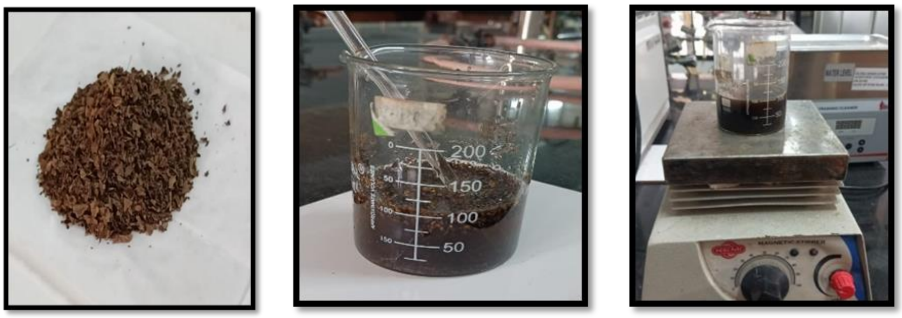
3.3 PREPARATION OF ALOE-VERA EXTRACT

Aloe vera leaves were collected from nearby garden and was cleaned and washed with tap water. The leaves were cut into small pieces using a knife and were dried in sunlight For 2 days. 6 g of dried leaves were weighed using an electronic balance and grinded to powdery form. The dried aloe-vera powder was mixed in 100ml distilled water in a beaker. The beaker with the solution was kept in oven at 100 °**C** for 20 minutes till the solution turns light yellow in colour. It was then stirred in a magnetic stirrer for 20 more minutes. The stirred solution was cooled to room temperature and filtered using a normal filter paper first and then using a Whatman filter paper (Grade No.40). The yellowish coloured clear solution obtained is the aloe-vera extract. It was stored in refrigerator at 4° C. The extract preparation is outlined in fig.3.2. This extract was then used for the synthesis of copper oxide (CuO) nanoparticles

ALOE VERA PLANT FROM GARDEN

ALOEVERA LEAVES CLEANED AND CHOPPED

CHOPPED ALOEVERA IS SUNDRIED



6g OF DRIED LEAVES IS POWDERED AND WEIGHED

DRIED ALOEVERA POWDER IS MIXED WITH 100ml DISTILLED WATER

THE SOLUTION IS STIRRED IN A MAGNETIC STIRRERFOR 20 MINS AT TEMP 60°C

COOLED SOLUTION IS FILTERED BY WHATMANN 40 FILTER PAPER

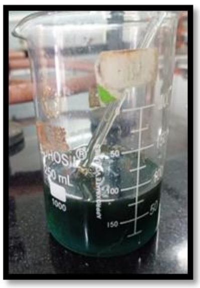
YELLOW CLEAR ALOEVERA EXTRACT

Fig.3.2. preparation of aloe vera extract

3.4 SYNTHESIS OF COPPER OXIDE NANOPARTICLES

In order to synthesize copper oxide nanoparticles, the copper precursor used is $CuSO_4.5H_2O$. As the first step 1 M copper sulphate solution is prepared, for that 12.48 g of $CuSO_4.5H_2O$ is dissolved in 50 ml of distilled water.15 ml of prepared aloe-vera extract was mixed with The 1 M copper sulphate solution (12.48g+50 ml) in a beaker. It was then stirred for 30 Minutes using a magnetic stirrer at 100°C. It was then kept for 45 min an oven at 100°C. The prepared green colour solution was maintained for 72 hours. 60 g NaOH pellets were weighed using an electronic balance and was dissolved In 50 ml distilled water to prepare 15 M NaOH solution. This aqueous NaOH solution was added drop by drop to the prepared solution from a beurette with continuous stirring using a magnetic stirrer. The colour of the prepared solution precipitated and changes colour from green to blue, then to brown and finally to a brownish black. The solution was stirred continuously for 30 more minutes after the precipitate was formed to make sure that no precursor particles remain unreacted. The thick solution was then transferred to centrifuging tubes and centrifuged at 1300 rpm for 10 minutes. The supernatant was removed and the precipitate was washed thoroughly with distilled water and again centrifuged and the process was repeated till all the NaOH was washed off from the precipitate. The precipitate was then carefully decanted from the centrifuging tube to a watch glass using a micro spatula. The precipitate in watch glass was dried in oven at 80°C till all the water content was evaporated from it. It took about 30 minutes to get dried. The dried precipitate was carefully scraped off from the watch glass and was finely powdered. This fine brownish black powder obtained is the copper oxide nanoparticles and was stored in an airtight sample bottle and was given for further characterization.

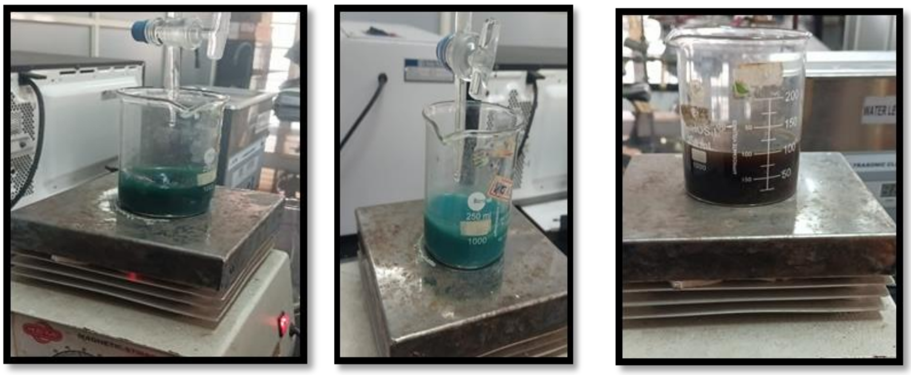
1 M COPPER SULPHATE SOLUTION IN DISTILLED WATER



ALOEVERA EXTRACT MIXED WITH COPPER

STIRRED FOR 30 MINS IN MAGNETIC STIRRER

PREPARED 10M Aq.NaOH SOLUTION



COLOUR OF PREPARED SOLN PRECIPITATED CHANGES FROM GREEN TO BLUE AND FINALLY TO BROWNISH BLACK

THICK SOLN IS CENTRIFUGED AT 1300RPM FOR 10MINS

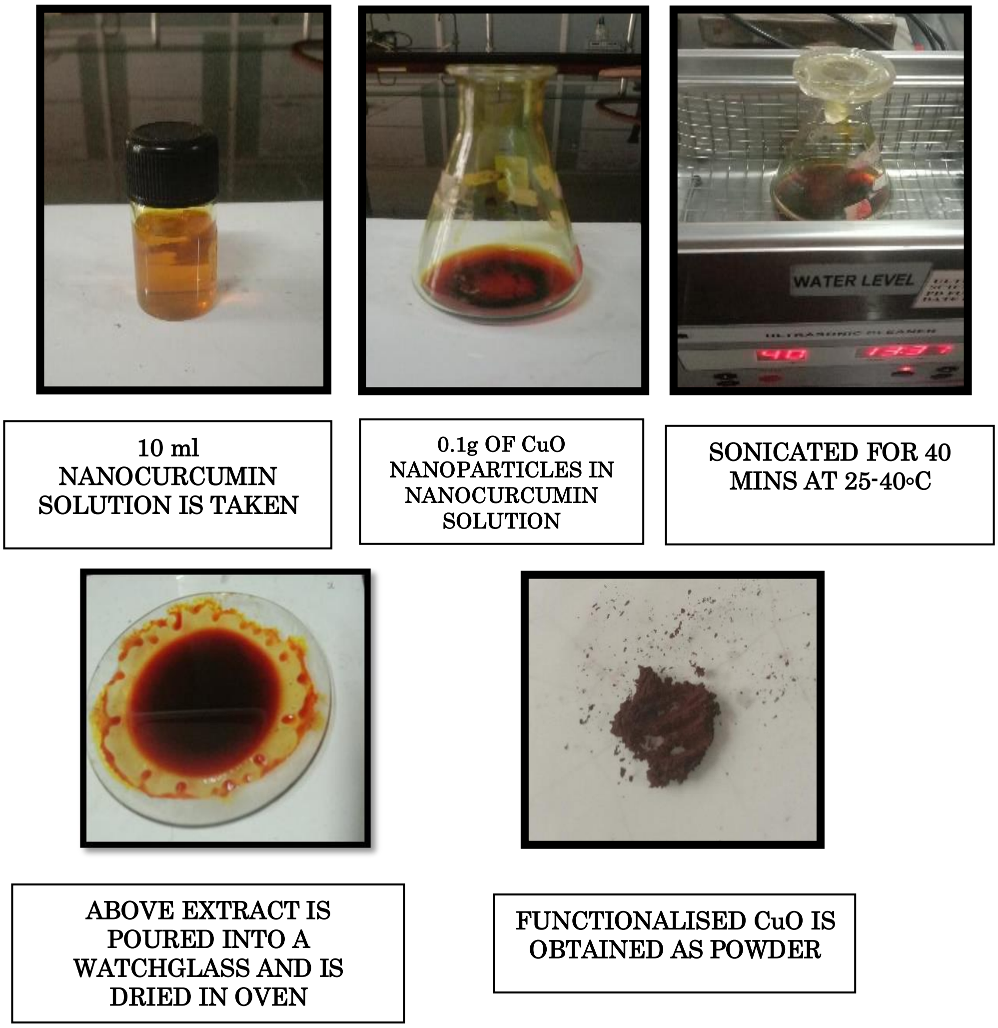
THE PRECIPITATE AFTER WASHING IS THEN POURED IN A WATCH GLASS AND DRIED IN OVEN

DRIED PRECIPITATE WAS CAREFULLY SCRAPED OFF FROM THE WATCH GLASS AND CuO NANOPARTICLES IS OBTAINED AS FINE POWDER

Fig 3.3 Synthesis route of CuO nanoparticles

3.5 FUNCTIONALISATION OF SYNTHESISED COPPER OXIDE NANOPARTICLES NANOPARTICLES WITH NANOCURCUMIN.

About 0.1g of synthesised CuO nanoparticles was dissolved in 10 ml of nanocurcumin solution taken in 50 ml conical flask. It is then ultrasonicated in an ultrasonic bath. The sonication was performed for 40 min within the temperature range 25-40°C.The obtained extract was poured into a watch glass and dried in an oven. The dried precipitate was carefully scraped off from the watch glass and was finely powdered. This fine brownish black powder is nano curcumin functionalised CuO nanoparticles. It was stored in an airtight sample bottle and was given for further characterization.



10 ml NANOCURCUMIN SOLUTION IS TAKEN

0.1g OF CuO NANOPARTICLES IN NANOCURCUMIN SOLUTION

SONICATED FOR 40 MINS AT 25-40°C

ABOVE EXTRACT IS POURED INTO A WATCHGLASS AND IS DRIED IN OVEN

FUNCTIONALISED CuO IS OBTAINED AS POWDER

Figure 3.4 Synthesis route of nanocurcumin functionalised CuO nanoparticles

3.6 ADSORPTION STUDIES OF DYES USING NANO CuO AND NANO CURCUMIN FUNCTIONALISED NANO CuO

Dye adsorption studies of CuO nano particles and nano curcumin functionalized CuO nanoparticles were carried out by using Eriochrome Black-T, methyl orange and methylene blue as model dyes. For the study ,about 15 ppm of each dye solution was prepared by weighing 0.01 g of each dye into 100 ml standard flask and madee up to the volume using distilled water.Then pipette out 15 ml of this solution.The pipetted solution is made up to100 ml and this is 15 ppm of each dye solution. 20 ml of the above solution is taken in two separate beakers and add 0.1 g of CuO nanoparticles in one beaker and 0.1 g of nano curcumin functionalised CuO nanoparticles in the other. These solutions were stirred for about 1 hr and it was maintained for a day. Then solution was centrifuged.The supernatant solution was clear. This confirmed its application in dye adsorption.For eachcase the adsorption percentage was analysed uv -visible spectroscopy.

3.7 ANTIBACTERIAL STUDY

Agar well diffusion method is widely used to evaluate the antimicrobial activity of plants or microbial extracts.For this solutions of antimicrobial agent was prepared by dissolving a)0.1g CuO nanoparticles in 10 ml acetone and b)0.1 g of nanocurcumin functionalised CuO nanoparticles in 10 ml acetone by employing ultrasonication method. Then agar plate surface is inoculated by spreading a volume of the microbial inoculum [e.coli] over the entire agar surface. Then, a hole with a diameter of 6 to 8 mm is punched aseptically with a sterile cork borer or a tip, and both volume (20–100 µL) of the antimicrobial agent at desired concentration is introduced into the well. Then, agar plates are incubated under suitable conditions depending upon the test microorganism. The antimicrobial agent diffuses in the agar medium and inhibits the growth of the microbial strain tested. The antibacterial activities of both samples were compared.

Chapter 4

Results and discussion

4.1. CHARACTERISATION OF NANOCURCUMIN

Nanocurcumin was synthesized using a previously reported ultrasonication method. Synthesized nanocurcumin was characterized using UV-visible absorption spectroscopy, FT-IR spectroscopy and Scanning Electron Microscopy.

4.1.1 UV-VISIBLE ABSORPTION SPECTROSCOPY

UV-visible absorption spectrum of synthesised nanocurcumin was taken in acetone and is depicted in Fig.4.1.The spectrum showed a broad absorption peak centered at 420 nm which is the characteristic peak of pure nanocurcumin as ireported by Mohamed et al(2020)[32].This confirmed the formation of nanocurcumin [43].

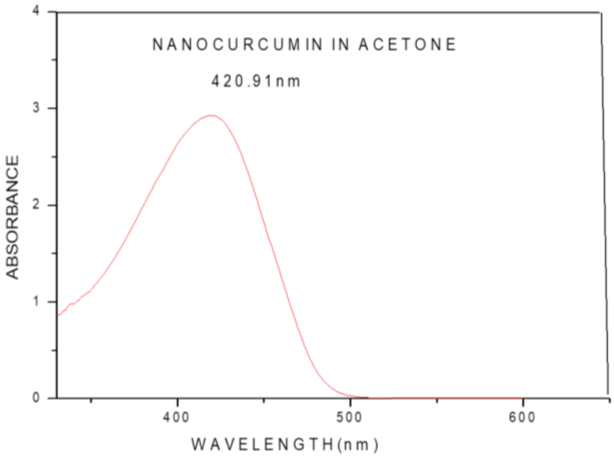


Figure 4.1 UV spectrum of nanocurcumin

4.1.2 FOURIER TRANSFORMED INFRARED (FT-IR) SPECTROSCOPY

The synthesized nanocurcumin is subjected to FTIR analysis at room temperature to confirm the formation of nanocurcumin (Fig.4.2).An intense band at 3413 cm^{-1} is due to stretching vibrations of hydrogen bonded OH group.The presence of bands at 1637 cm^{-1} and 1596 cm^{-1} indicates C=O strectching frequency and stretching vibrations of C-C respectively. The observed peak at 1460 cm^{-1} corresponds to aromatic stretching vibrations of benzene ring.The bands at 2921cm^{-1} and 2852 cm^{-1} originate from the asymmetric stretching vibrations of Csp^{2}–H and Csp^{3}–H bonds. The band centered at 1268 cm^{-1} originates from the stretching vibrations of Csp^{2}– O bonds. The observed bands are comparable to that obtained as per the study by Hettiarachchi et al.(2021).This again confirmed the formation of nanocurcumin. [44]

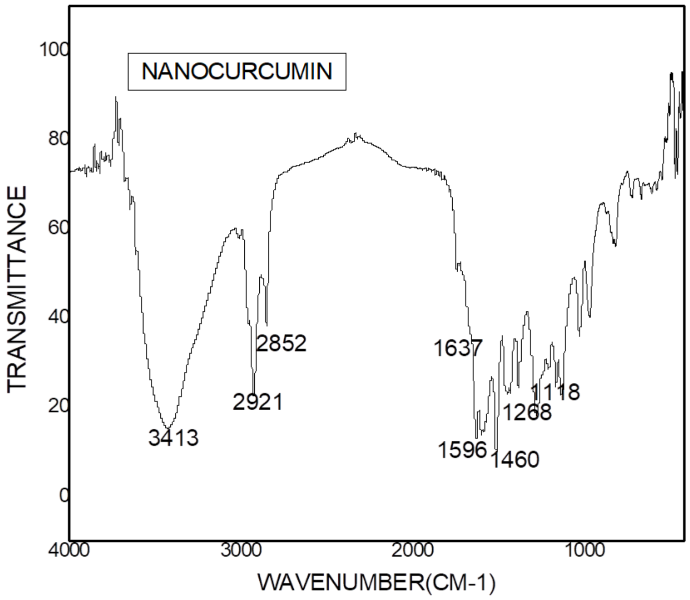


Figure 4.2 FTIR spectrum of nanocurcumin

4.1.3 SCANNING ELECTRON MICROSCOPIC (SEM) ANALYSIS

The SEM images showed uniformly distributed curcumin nanoparticles having size in the range 100-170nm as reported by Varun Kumar et al.(2021).It indicates the successful preparatrion of curcumin nanoparticles.[45].The SEM images of synthesised nanocurcumin are shown in fig 4.3 and fig 4.4.

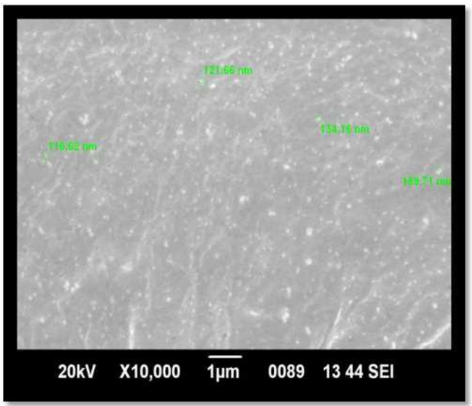


Figure 4.3(a) SEM images of nanocurcumin 1µm

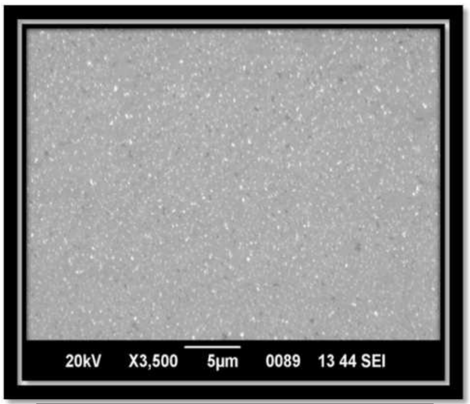


Figure 4.3(b) SEM images of nanocurcumin

4.2 CHARACTERISATION OF CuO NANOPARTICLES AND NANOCURCUMIN FUNCTIONALISED CuO NANOPARTICLES

4.2.1 UV VISIBLE- ABSORPTION SPECTROSCOPY

UV-Visible absorption spectrum of synthesied CuO nanoparticles (CuO) and nanocurcumin functionalised CuO nanoparticles (CuO-NC) were taken in acetone and are depicted in fig 4.4 and fig 4.5 respectively. The UV-Visible absorption spectrum of CuO showed the characteristic peak of CuO nanoparticles at 339 nm and confirmed its formation. In the UV-visible absorption spectrum of CuO-NC there is a peak at 325 nm due to CuO nanoparticles and a peak at 418 nm due to the presence of Curcumin nanoparticles. Both peaks are shifted, which confirmed the functionalisation.

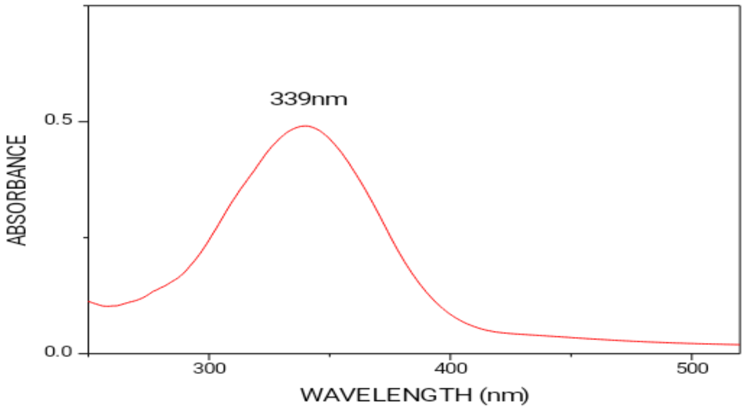


Figure 4.4 UV absorption spectrum of CuO

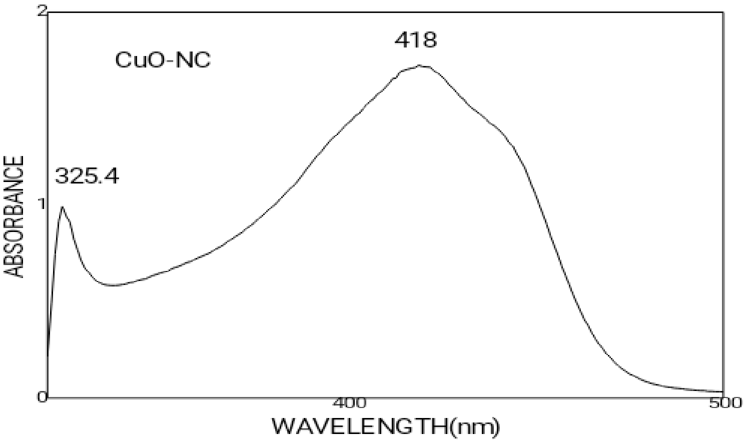


Figure 4.5 UV absorption spectrum of CuO-NC

4.2.2 FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY

The synthesized CuO nanoparticles were analysed using FTIR analysis at room temperature to confirm the formation of CuO nanoparticles. An intense band at 3427 cm^{-1} was attributed to the O–H stretching vibration of surface hydroxyl groups of adsorbed water molecules which arises because nanocrystalline materials having a high surface-to-volume ratio absorbs high moisture. The presence of band 1000cm^{-1} indicates bending vibration of the Cu– O bond. The observed peaks at 1643 cm^{-1} and 603 Cm^{-1} corresponds to the characteristics stretching vibration of Cu-O bond in the CuO.

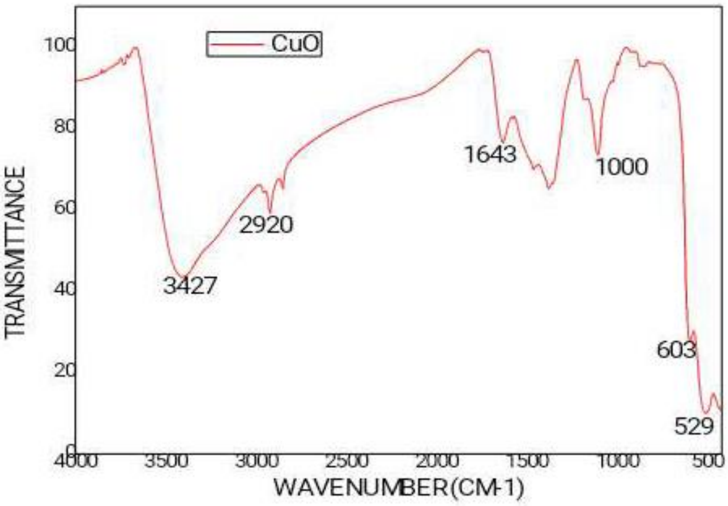


Figure 4.6 FTIR spectrum of CuO nanoparticles

The FT-IR spectrum of nanocurcumin (Fig 4.2) and CuO-NC (Fig 4.7.) were taken and analysed. The O-H stretching of nanocurcumin is shifted from 3413cm^{-1} to 3400 cm^{-1} in functionalised sample. Also the C=O stretching frequency at 1637 cm^{-1} is coupled with Cu-O stretching and shifted to 1603 cm^{-1}.The peak at 1118 cm-1 due to C-O stretching in CuO is also shifted to 1153 cm^{-1}.This confirmed the functionalisation of CuO with curcumin at O-H and C=O positions**.**

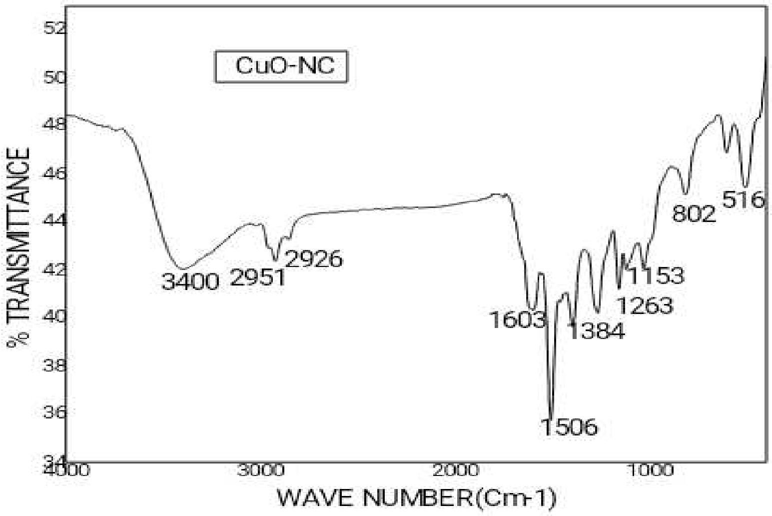


Figure 4.7 FTIR spectrum of CuO-NC

4.2.3 X-RAY DIFFRACTION (PXRD) STUDY

4.2.3.1 XRD OF CuO NANOPARTICLES

The phase purity and structural characteristics of the synthesized CuO nanosheets were examined using XRD analysis (Fig 4.8). The XRD Diffraction patterns appeared at 2ɵ (32.54◦,35.56◦,38.77◦,48.74◦,58.37◦,61.56◦and 68.17◦ values and they were assigned to the corresponding (110),(002),(111),(020),(013),(310) and (113).The reflection peaks were identical to the single-phase CuO with a monoclinic structure and the diffraction data corresponded to the JCPDS card of CuO (JCPDS 80-1268). No peaks of impurity were observed in the XRD pattern.

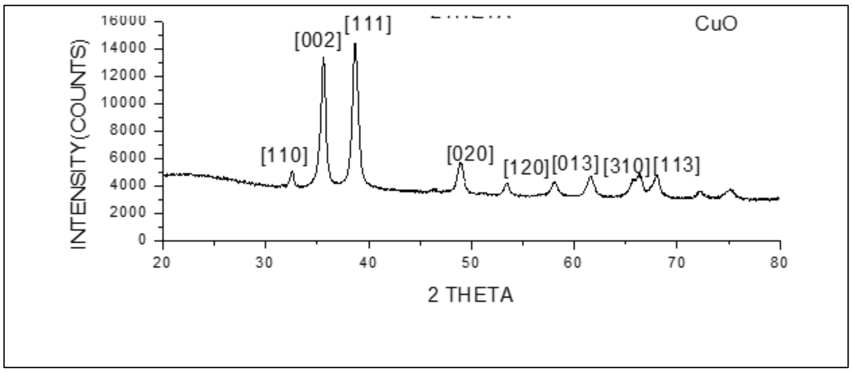


Figure 4.8XRD spectrum of CuO nanoparticles

The particle size was calculated for both samples using Debye Scherrer equation.

$$\mathbf{D = K\Lambda / \beta COS\theta}$$

Where β is the full width at half maximum (FWHM),θ is the angle of the maximum peak, Λ is the wavelength of the source, D Is the size of the particle and K is the scherrer constant.

AVERAGE PARTICLE SIZE OF CuO NANOPARTICLES

PEAK 1

$2\theta=32.543°=0.567982498$ rad

$\theta= 0.2839912492$ rad

β=0.282=0.0049218285 rad

Λ=1.5406×10-10

From Debye Scherrer equation

$$D = \frac{0.890\times1.5406\times10\text{-}10m}{0.0049218285\times\cos\ (0.283991492)}$$

=2.902065238×10—8m

=29.02065238×10-9m

=29.020652 nm

PEAK 2

2θ=35.602°

θ=17.8005°=0.3106773335rad

β=0.507°=0088488193

Λ=1.5406×10-10m

K=0.890

$$D = \frac{0.890\times1.5406\times10\text{-}10}{0.0088488193\times\cos\ (0.3106773335)}$$

=16.27420634×10-9m

=16.27420634 nm

PEAK 3

$K=0.890$

$\beta =0.653° = 0.011397$ rad

$2\theta=38.691°$

$\theta=19.3455°=0.3376426704$ Rad

$\Lambda=1.2506\times10\text{-}10m$

$$D = \frac{0.890\times(1.5406\times10\text{-}10m)}{0.011397\times\cos (0.3376426704)}$$

$=1.275057797\times10\text{-}8m$

$=12.75057797\times10\text{-}9m$

$=12.75057797$ nm

PEAK 4

$K=0.890$

$B=0.579=0.010105456$ rad

$2\theta=49.031°$

$\theta=24.5155° = 0.4278761928$ rad

$\Lambda=1.5406\times10\text{-}10m$

$$D = \frac{0.890\times (1.5406\times 10-10)}{0.010105456\times \cos (0.4278761928)}$$

$= 0.1491264356\times 10-8m$

$=14.91264356\times 10-9m$

$=14.91264356$ nm

The average particle size is found to be 14.64580929 nm.

4.2.3.2 XRD OF NANO-CURCUMIN FUNCTIONALISED CuO NANOPARTICLES

XRD patterns when compared with standard JCPDS file number(80-1268). The data revealed the formation of Cuo nanostructure in monoclinic symmetry. No extra peaks were observed, pointing to single plane structure. The functionalised sample showed more diffraction peaks and this is due to its grain shape. The charachteristic peaks at 35.58°, 38.68°, 49.12°, 61.52° are assigned to the (110), (002), (202) and (013) which represents the reflection lines of monoclinic CuO nanoparticles[45].

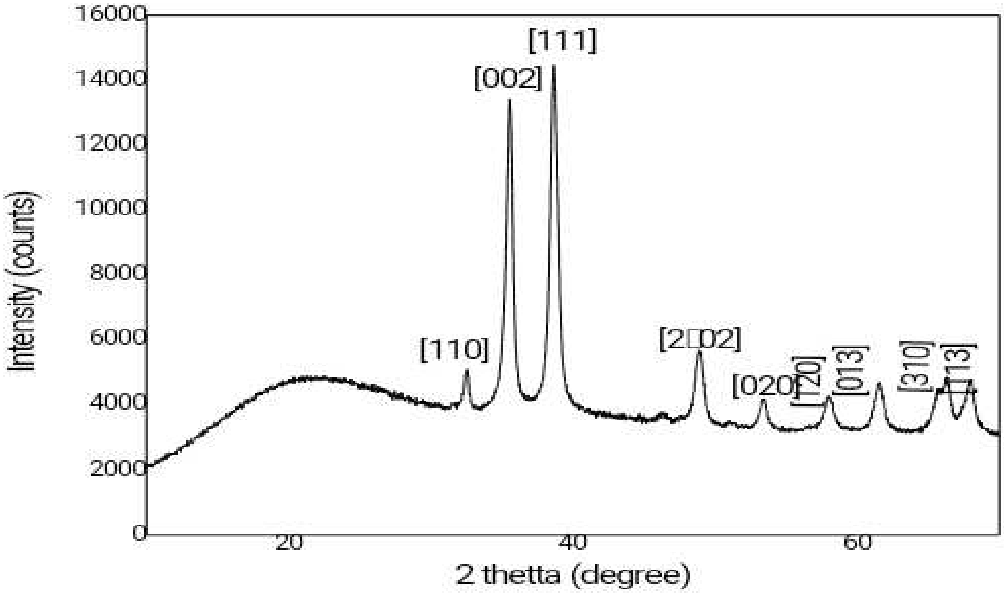


Figure 4.9 XRD spectrum of CuO-NC

The average particle size of CuO –NC nanoparticle is calculated as 143.15 nm. So the functionalized sample has an increased particle size than bare CuO.

In the XRD spectrum of nanocurcumin functionalised copper oxide nanoparticles, the broad peak at about 21 degree is the characteristic peak of nanocurcumin. All other peaks corresponds to the monoclinic CuO nanoparticles.

4.2.4 SCANNING ELECTRON MICROSCOPY(SEM)

The SEM images of the synthesized CuO and nanocurcumin functionalised CuO nanoparticles are given below.

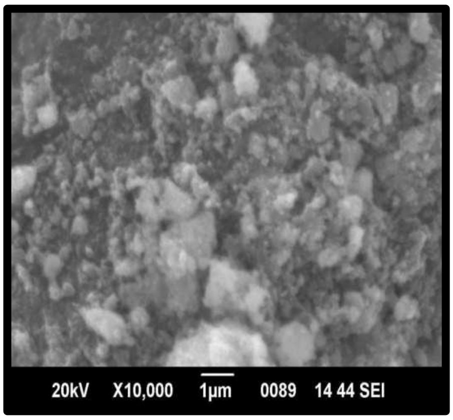


Figure 4.10 SEM images of CuO nanoparticles 1 µm

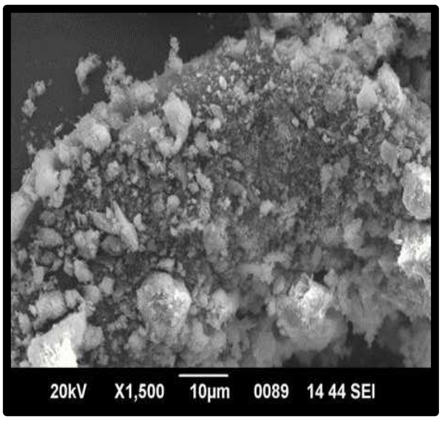


Figure 4.11 SEM images of CuO nanoparticles 10 µm

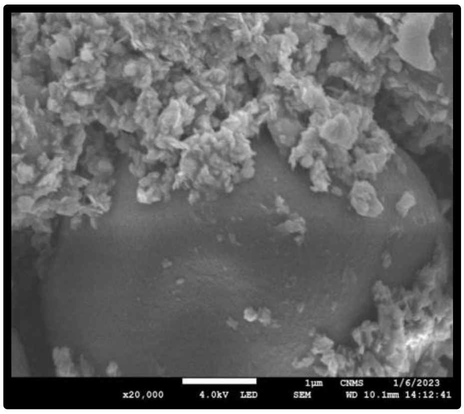


Figure 4.12 SEM images of CuO NC

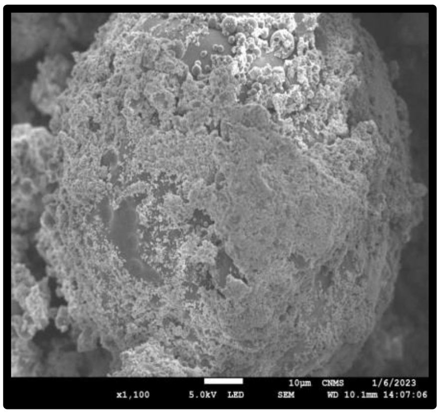


Figure 4.13 SEM images of CuO NC

The SEM images shows that particles are less agglomerated in CuO-NC and have more uniform distribution when compared to bare CuO.

4.2.5 ENERGY DISPERSIVE SPECTRUM (EDS) ANALYSIS

The EDS spectrum of both CuO and CuO-NC contains peaks of Cu and O only. It is confirmed that both samples are pure without any impurity. CuO showed 43.74 %.Cu and 56.26% O. So obtained sample is CuO. Where as in CuO-NC the composition of oxygen is more (70.16%) than Cu(29.84%) . The increased atomic percentage of oxygen in CuO-NC is due to the presence of excess oxygen via functionalization. It confirmed the functionalisation.

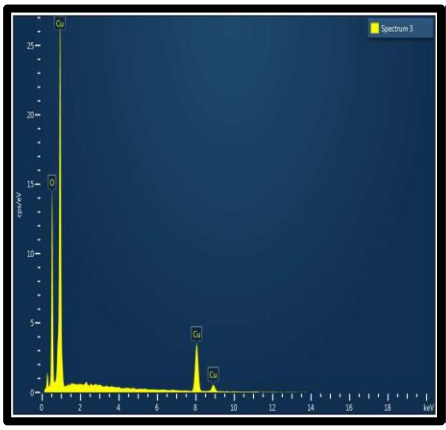


Figure 4.14 EDS spectrum of CuO Nanoparticles

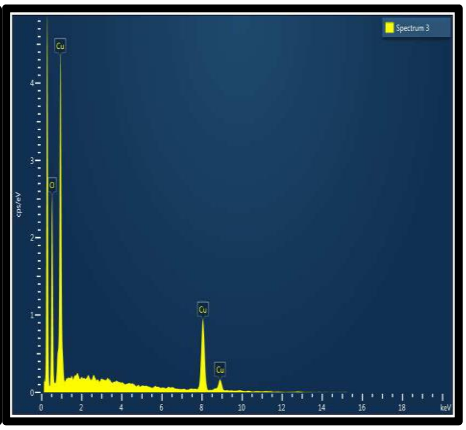


Figure 4.15 EDS spectrum of CuO-NC

ELEMENT	LINE TYPE	Wt%	ATOMIC %
O	**K SERIES**	**24.47**	**56.26**
Cu	**K SERIES**	**75.53**	**43.74**
TOTAL		**100**	**100**

Table 4.1Atomic% and Wt% of CuO

ELEMENT	**LINE TYPE**	**WT%**	**ATOMIC %**
O	**K SERIES**	**37.18**	**70.16**
Cu	**K SERIES**	**62.28**	**29.84**
TOTAL		**100**	**100**

Table 4.2 Atomic and Wt% of CuO-NC

4.2.6 TRANSMISSION ELECTRON MICROSCOPY(TEM)

TEM images of nanocurcumin functionalised CuO nanoparticles are given below. The SAED pattern revealed the polycrystalline nature of nanocurcumin functionalized CuO-nanoparticles. HRTEM images with d-spacing 0.28 nm indicates(110) plane**.** Sheet like morphology of nanocurcumin functionalised CuO nanoparticles is also confirmed from the TEM images.

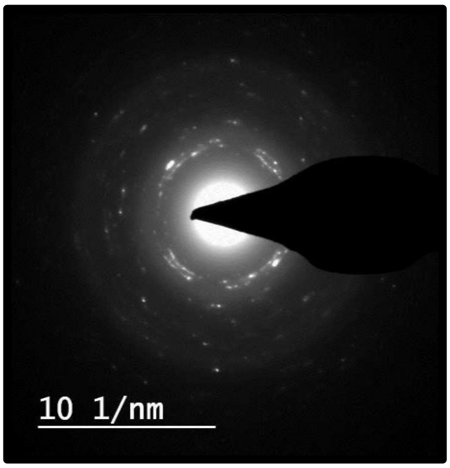


Figure 4.16 SAED pattern of CuONC

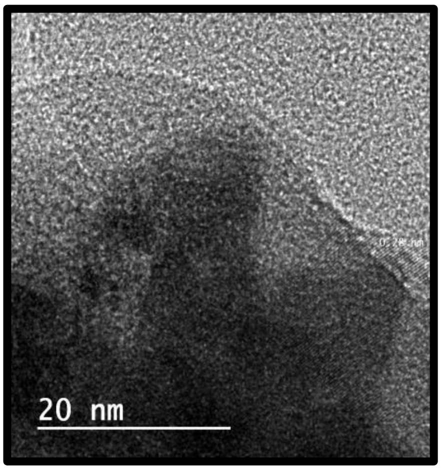


Figure 4.17 HRTEM image of CuONC

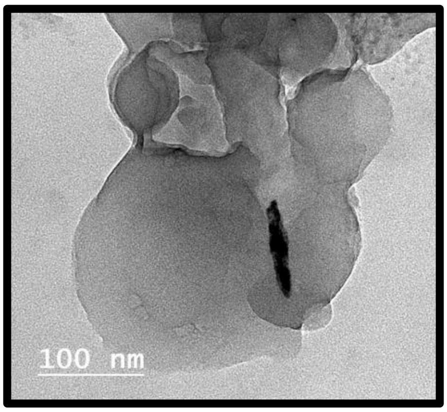


Figure 4.18 HRTEM image of CuONC 100nm

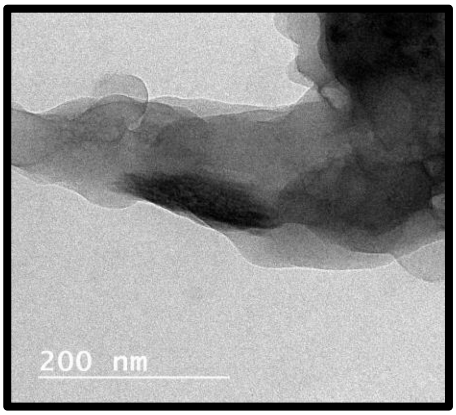


Figure 4.19 HRTEM image of CuONC 200nm

4.2.7 BRUNAUER-EMMETT-TELLER (BET) ANALYSIS OF COPPER OXIDENANOPARTICLES:

Adsorption/Desorption isotherm and BJH plot of Copper oxide nanoparticles are shown in the figures respectively. The surface area, pore volume and pore diameter of cop peroxide nanoparticles were calculated as 31.026 m^2/g, 0.1769 cm^3/g and 9.46 nm from BJH plot.

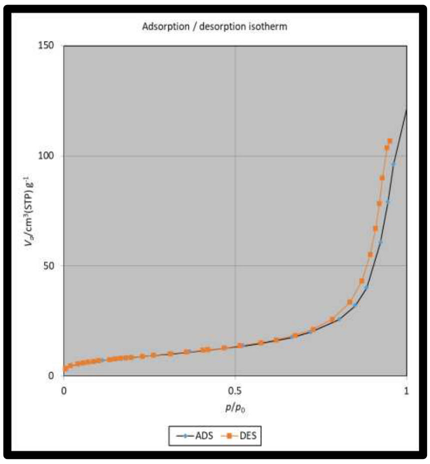


Figure 4.20 Adsorption/Desorption Isotherm of CuO

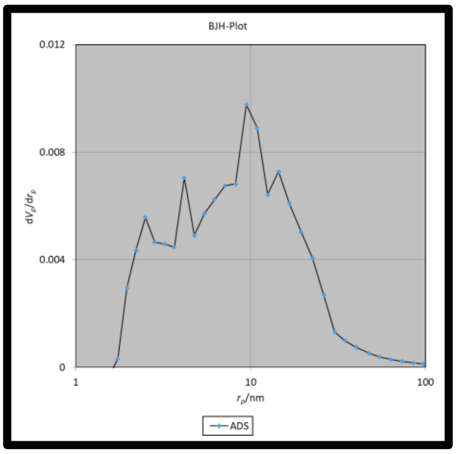
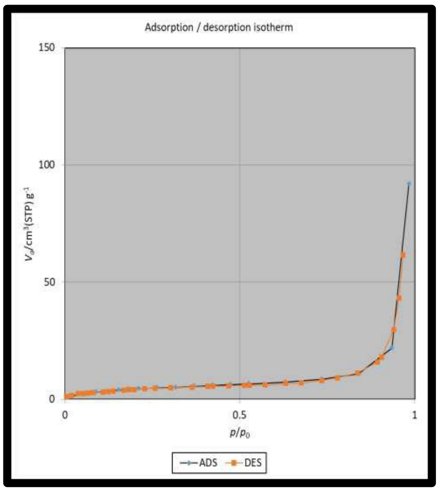


Figure 4.21 BJH plot of CuO

BRUNAUER-EMMETT-TELLER (BET) ANALYSIS OF NANOCURCUMIN FUNCTIONALISED CuO NANOPARTICLES:

Adsorption/Desorption isotherm and BJH plot of functionalised nanocurcumin copper oxide nanoparticles are shown in the figures respectively. The surface area, pore volume and pore diameter of functionalised copper oxide nanoparticles were calculated as 14.348m^2/g,0.1375 cm^3/g and 19.38 nm.

On assessment it was observed that, the pore diameter of CuO-NC increased due to the functionalisation and this resulted in the decrase in pore volume and surface area.



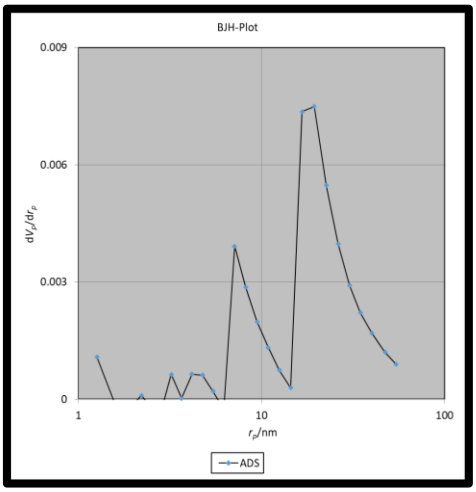


Figure 4.22 Adsorption/Desorption isotherm of CuO-NC

Figure 4.23 BJH plot of CuO-NC

4.4 DYE ADSORPTION STUDY

Dye adsorption studies of CuO nanoparticles and nanocurcumin functionalized copper oxide nanoparticles were done using cationic and anionic dyes such as Eriochrome Black-T, methyl orange and methylene blue as model dyes by employing UV-visible spectroscopy.

4.4.1 DYE ADSORPTION STUDY USING METHYL ORANGE

Two peaks were obtained for Methyl orange at about 250 and 450 nm due to $\pi \rightarrow \pi^*$ and n $\rightarrow \pi^*$transitions, respectively. The peak intensity were reduced after adsorption on CuO nanoparticles and nanocurcumin functionalized copper oxide nanoparticles which confirmed the methyl orange removal from water by adsorption by using both samples. The methyl orange adsorption percentage was found to be 38.97% for CuO after 60 minutes of stirring while it was 47.52% for CuO-NC at the same time. Thus better efficiency of CuO-NC for dye adsorption was confirmed.

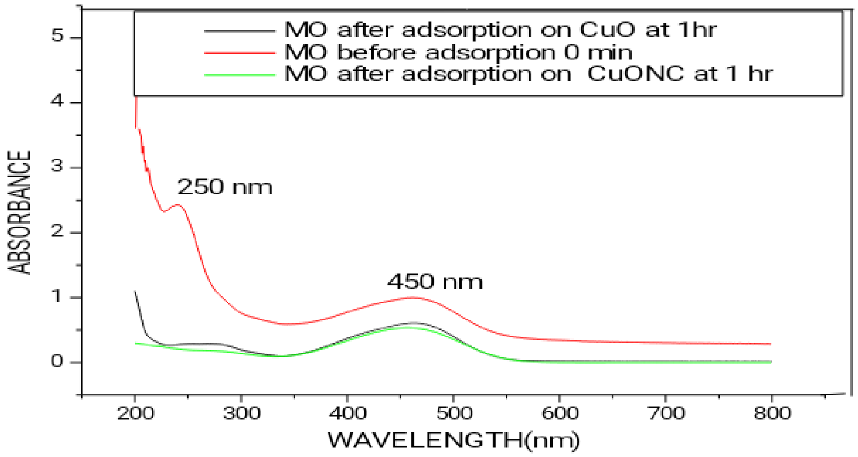


Figure 4.24 UV-visible spectrum of dye adsoption study using methyl orange.

4.4.2 DYE ADSORPTION STUDY USING EBT

Two peaks were obtained for Methyl orange at about 350 and 560 nm due to π→π* and n →π*transitions, respectively. The peak intensities were reduced after adsorption on CuO nanoparticles and nanocurcumin functionalized copper oxide nanoparticles which confirmed the EBT removal from water by adsorption. The EBT adsorption percentage was found to be 14.4% for CuO after 60 minutes of stirring while it was 22.48% for CuO-NC at the same time. Thus better efficiency of CuO-NC for dye adsorption was confirmed.

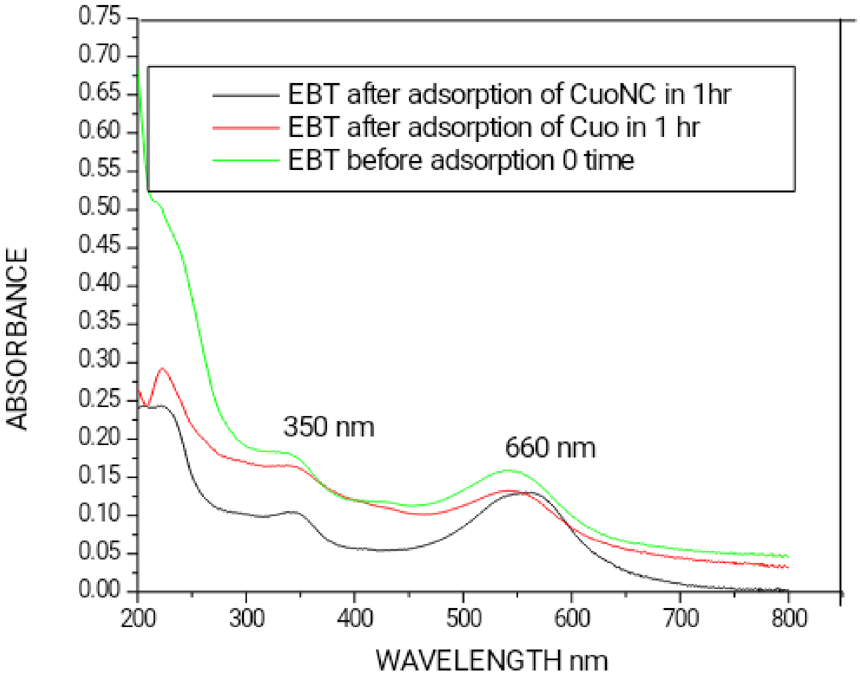


Figure 4.25 UV-visible spectrum of dye adsoption study using EBT.

4.4.3 DYE ADSORPTION STUDY USING METHYLENE BLUE

Two peaks were obtained for Methylene blue at about 290 and 660 nm due to $\pi \rightarrow \pi^*$ and n $\rightarrow \pi^*$transitions, respectively. The peak intensity were reduced after adsorption on CuO nanoparticles and nanocurcumin functionalized copper oxide nanoparticles which confirmed the methylene blue removal by adsorption.The methylene blue adsorption percentage was found to be 70.49% for CuO after 60 minutes of stirring while it was 95.56% for CuO-NC at the same time. Thus better efficiency of CuO-NC for dye adsorption was confirmed.

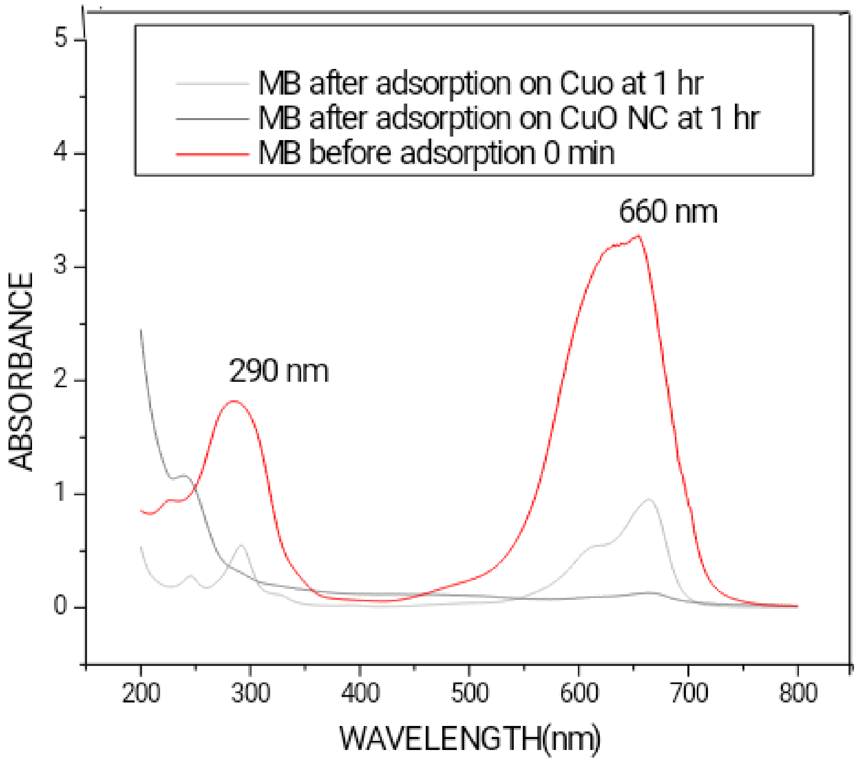


Figure 4.26 UV-visible spectrum of dye adsoption study using methylene blue.

4.3 ANTIBACTERIAL STUDY

The antibacterial activity of CuO nanoparticles and nanocurcumin functionalised CuO nanoparticles was tested against E.Coli bacteria using well diffusion method. In acetone, Cuo-NC showed a better antibacterial activity against E.Coli when compared to bared CuO nanoparticles. The zone of inhibition is given in table 4.3 and the photograph is given.

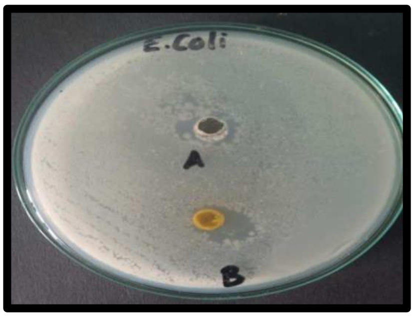


Figure 4.27 Antibacterial activity of CuO(A) and CuO-NC(B) against E.Coli.

BACTERIA	ZONE OF INHABITATION(mm)	
	CuO	CuO-NC
E.COLI	8	12

Table 4.3 Zone of inhibition of CuO and CuO –NC against E.Coli.

Chapter 5

Conclusion

According to the concept of green chemistry, we designed a method to develop nanocurcumin functionalized CuO nanoparticles. Green synthesis of nanocurcumin was done from curcumin rhizomes by ultrasonication. The synthesized nanocurcumin was characterised using UV- visible absorption spectroscopy, FTIR spectroscopy and SEM analysis. Further, green synthesis of CuO nanoparticles and nanocurcumin functionalised CuO nanoparticles were done using aloe-vera extract and copper sulphate as precursor. The synthesized nanoparticles were characterized using UV-visible absorption spectroscopy, FTIR spectroscopy, PXRD, SEM, EDS,TEM and BET analysis.The average particle size of CuO nanoparticles and nanocurcumin functionalised CuO nanoparticles were calculated from PXRD as 14.64nm and 143.15nm,respectively.SEM and TEM images of prepared nanoparticles indicates homogenous distribution of nanoparticles and polycrystalline nature. FTIR spectroscopy and UV-visible absorption spectra confirmed the functionalization of CuO by nanocurcumin. The antibacterial activity of synthesised CuO nanoparticles and nanocurcumin functionalised CuO nanoparticles was studied and the results indicated that nanocurcumin functionalised CuO nanoparticle showed a better activity than bare CuO against E-Coli. Dye adsorption abilities of bare CuO and functionalised sample was carried out using various cationic and anionic dyes such as EBT (anionic dye), methylene blue (cationic dye) and methyl orange (anionic) as model dyes. The results demonstrated that the functionalized CuO nanoparticles are better dye adsorption agents due to it’s increased pore diameter. It was evaluated that 47.52%, 22.48% and 95.56% of Methyl orange, EBT and Methylene blue, respectively, were adsorbed on nanocurcumin functionalized CuO-nanoparticles within 60 minutes. which is far better that the adsorption percentage using CuO nanoparticles adsorption percentage at the same time (38.97% , 14.4%, 70.49% for methyl orange, EBT and Methylene blue, respectively). Hence the nanocurcumin functionalized CuO nanoparticles can be used as multifunctional material against bacterial infections and also for water purification.

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