HYDROTHERMAL SYNTHESIS AND CHARACTERIZATION OF PEG CAPPED COPPER OXIDE NANOPARTICLES

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BHARATA MATA COLLEGE

THRIKKAKARA

CERTIFICATE

*This is to certify that the project report entitled "**HYDROTHERMAL SYNTHESIS AND CHARACTERIZATION OF PEG CAPPED COPPER OXIDE NANOPARTICLES**" is a bonafide work carried out by **SRUTHY MOL P.B** , M.sc Pharmaceutical Chemistry ,under my supervision and guidance and that no part of this has been submitted for any degree, diploma or other similar titles of recognition under any university*

Forwarded by project guide

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DECLARATION

I, **SRUTHY MOL P.B** hereby declare that this project report entitled ***"HYDROTHERMAL SYNTHESIS AND CHARACTERIZATION OF PEG CAPPED COPPER OXIDE NANOPARTICLES"*** is an authentic work carried out during my course under the guidance of Dr. JINSA MARY JACOB , Department of Chemistry , Bharata Mata College Thrikkakara

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Key words : CuO nanoparticles,hydrothermal method, PEG as capping agent

ABSTRACT

Scientific Research on Nanoparticles Proves that they have many potential application_in preclinical , clinical Medicine , Physics, Optics ad Electronic. Metal oxide Nanoparticlesb such as Copper oxide (CuO) have attracted attention mostly because of their antimicrobial and biocide properties and also their biomedical application .H ere in this study Hydrothermal Method was used to synthesis copper oxide nanoparticles and its characterization . The PEG capped CuO nanoparticles was synthesized from copper acetate monohydrate by hydrothermal method within a stainless steel autoclave .The synthesiszed CuO nanoparticles is characterized by infrared spectroscopy (IR), ultraviolet spectroscopy (UV) and powder X-Ray diffraction (powder XRD). Copper oxide nanoparticles due to their vast application prominently used in various field. They have greater biological properties including effective antimicrobial action against a wide range of pathogen and also drug resistant bacteria.

Keywords: hydrothermal method , copper acetate monohydrate , polyethylene glycol

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INTRODUCTION

Since the last ten years, there has been an increasing media and public interest in nanotechnology due to its quick development and expansion, which has numerous uses in numerous research, development, and industrial activities. All of these programs have the development of novel nanomaterials as a central subject [1].Nanotechnology involves the production of specific structures by manipulating individual atom , or molecule as well as the material which contain very small structures. Nanoparticle is the main outcome of nanotechnology . Nanoparticles is defined as smaller than 100 Nanometers at least in one dimension. Even though they share the same chemical compostion as well-known materials in bulk form, nanomaterials may behave differently when ingested into the body due to differences in their physico-chemical characteristics. Thus, they might provide various potential hazards [2].

NANOMATERIALS also known as nanocrystalline materials, have grains that are about a billionth of a meter wide. These materials can be worked with to achieve a wide range of structural and non-structural purposes because of their incredibly beneficial and interesting qualities. A nanomaterial has grains that are between 1 and 100 nm in size. One atom typically measures between one and two angstroms (Å) in radius. Since one nanometer comprises 10 atomic radii, it is most likely to contain three to five atoms. At high temperatures, nanocrystalline materials are incredibly strong, ductile, and hard. They are also extremely chemically active and resistant to corrosion, wear, and erosion.When compared to the conventional substitutes that are now on the market, nanomaterials, or nanocrystalline materials, are also far more formable. In the middle of the 1980s, the study of nanomaterials literally took off in the United States[3]. For the synthesis of nanomaterials, top-down and bottom-up techniques are typically used. In top-down methods the bulk materials are divided to create nanostructured materials using a mining process. Mechanical milling, laser ablation, etching, sputtering, and electro-explosion are examples of top-down techniques.In bottom -up approaches include spinning, laser pyrolysis, chemical vapour deposition, chemical reduction, green synthesis, molecular condensation, sol gel process, supercritical fluid synthesis[4]. Nanostructures are made up by chemical processes in the bottom -up approaches. The choice for choosing the appropriate appropriate procedure is depend on the chemical makeup and the desired properties specified for the nanoparticles[5].

The use of nanotechnology and nanomaterials is usually found in these areas[6].

- Medicine: Some nanomaterials' characteristics make them perfect for enhancing early cancer or neurological disease diagnosis and treatment. They have the ability to selectively attack cancer cells without damaging healthy cells. Some pharmaceutical products, like sunscreen, have also benefited from the addition of nanoparticles.

- Environment: Some of its environmentally friendly applications include heavy metal nanofiltration systems, wastewater treatment with nanobubbles, and ion-based air purification. There are also nanocatalysts available to improve the effectiveness and reduce the pollution of chemical reactions.
- Textile: Nanotechnology enables the creation of stronger, lighter, and more durable materials for sports equipment and motorcycle helmets as well as intelligent fabrics that are wrinkle- and stain-resistant.
- Electronic: As a material for creating smaller, quicker, and more effective microchips and devices, carbon nanotubes are almost ready to take the place of silicon.Due to its characteristics, graphene is a great choice for flexible touchscreen development.
- Energy: A novel semiconductor created by Kyoto University enables the creation of solar panels that can convert two times as much sunshine into power. Additionally, nanotechnology decreases prices, creates stronger and lighter wind turbines, increases fuel economy, and, due to some nanocomponents' thermal insulation, can potentially save energy.
- Food: nanobiosensors might be used to identify infections in food, or nanocomposites could be utilized to boost food production by boosting mechanical and thermal resistance and reducing oxygen transfer in packed goods.

Nanoparticles are the main outcome of Nanotechnology.Nanoparticles are polymeric , spherical particles , composed of natural or synthetic polymers . These particles offer a wide range of possible uses because of their spherical form and high surface area to volume ratio[7]. Natural events generate nanoparticles, and many human industrial and household activities—including food preparation, the production of materials, and travel using internal combustion and jet engines—unintentionally release nanoparticles into the environment[8].

The core element of nanotechnology is nanoparticles (NPs). The term "nanoparticles" refers to particles with at least one dimension smaller than 100 nm. They could include metal, metal oxides, carbon, or organic substances [9].

Classification of nanoparticles based on the dimension

1. A zero-dimensional (0D) structure with a fixed length, width, and height. as in nano dot
2. One-dimensional (1D) structures with a single parameter. as in graphene
3. Two-dimensional (2D), which only has length and breadth as parameters. Eg, carbon nanotubes.
4. Three-dimensional (3D), having the three dimensions of length, width, and height. Eg,gold nanoparticles.

Based on the size,morphology,chemical and physical properties,nanoparticles are calssified into organic,inorganic and carbon based nanoparticles

- ### Organic nanoparticles

Organic nanoparticles are solid particles with a diameter between 10 nm to 1 m are made of lipids, polymers, or other organic substances. Common example of organic nanoparticles includes dendrimers,liposomes,micelles,ferritin etc. These organic NPs are more ideal for use in the biomedical area and are environmentally safe, biodegradable, non-toxic, and affordable.

- ### Inorganic nanoparticles

Particles formed of substances other than carbon are known as inorganic nanoparticles. It may include Metal and metal oxides . ie it can be two types

- ✓ **Metal based nanoparticles**: The transition metals were found to be the best candidates for the production of metal based NPs due to the presence of partally filled d orbitals which increase their redox activity.The size of the metal based NPs range from 10 to 100 nm.They found to be spherical and cylindrical shapes , among others. They have peculiar characteristics including high surface area to volume ratios, pore sizes, surface charge and density, crystalline and amorphous structures, high reactivity and sensitivity to environmental elements like air, moisture, heat, sunlight, etc. it can be obtained from metals such as,Al,Au,Ag,Cd,Co ,Cu,Fe,Pb,Zn

- ✓ **Metal oxide based nanoparticles** : metal based Nps which is converted to their respective oxides are termed as metal oxide based nanoparticles. Commonly used metal oxide nanoparticles include, copper oxide (CuO),titanium oxide (Tio), zinc oxide (ZnO),Aluminium oxide (Al_2O_3)

- **Carbon based nanoparticles :** Carbon-based NPs are the name given to nanoparticles made of carbon. Carbon-based NPs can be formed in a various shapes includes tubes, horns, spheres, and ellipses. Fullerene and carbon nanotubes (CNTs) are the two main groups of NPs based on carbon. Nanofibers, carbon black, and graphene are further kinds of carbon-based NPs.
  - ✓ **Fullerene** : There are several atomic clusters (Cn) in the fullerene family when n > 20. The most prevalent fullerene, fullerene C60, has 60 carbon atoms. It also known under the name "bucky ball." It has a spherical form. Each carbon atom is sp2 hybridized, and covalent bonds bind them all together. 20 hexagonal and 12 pentagonal vertices each contain one carbon atom. The spherical structure is made up of 28 to 1500 carbon atoms and has a diameter of up to 8.2 nm for a single layer and 4 to 36 nm for multi-layered fullerenes.
  - ✓ **Carbon nanotubes** : CNTs are cylindrical objects made of coiled graphene sheets and have a diameter of several nanometers. They can differ in length, diameter, symmetry, and layer count. CNT ends can either be closed by a half fullerene molecule or by being hollow. Due of their extraordinary rigidity, strength, and elasticity, CNTs have sparked significant economic interest. Additionally, they exhibit excellent electrical and thermal conductivity. They can be broadly divided into two groups based on their structural characteristics: (a) single-walled carbon nanotubes (SWCNTs), which have a diameter of 1-3 nm and a length of a few micrometers, and (b) multi-walled carbon nanotubes (MWCNTs), which have a diameter of 5-40 nm and a length of around 10 m. However, 550 nm-long CNTs have also been developed.
  - ✓ **Nanofibres** : carbon nanofibers (CNFs) can also produced by graphene sheets. . This graphene is organized into layers that are stacked like plates ,cups and cones. Excellent thermal, electrical and mechanical conductivity are all characteristics of CNFs. They range in diameter from 10 nm to 500

nm. As a result, these CNFs are used in a variety of industries, including photocatalysis,medicine delivery, sensors, energy devices,medicine delivery and nanocomposites.

- ✓ **Graphene** : Another allotrope of carbon is graphene. Its lattice resembles a two-dimensional honeycomb. Graphene sheet typically has a thickness of 1 nm.
- ✓ **Carbon black** : The amorphous materials known as carbon black nanoparticles (CBNP) or nano powders are mostly made of elemental carbon. 'Soot' or'shouen' are other names for it. These have a diameter between 20 and 70 nm and are spherical in form. Due to strong particle contact, CBNP aggregate into 500 nm-sized particles. These are typically used in laser printing and copier inks. They are also utilized in the plastics industry as colors and preservatives for rubber reinforcement.

The shape, size, and structure of the nanoparticles (NPs) can vary, including spherical, cylindrical, tubular, conical, hollow core, spiral, flat, and wire, among others. It may also have an uneven form. NPs can have a uniform or uneven surface. Additionally, they can exist as single- or multi-crystal solids in crystalline and amorphous forms. A multi-crystal solid may be agglomerated or loose. These NPs' variety in size & shape has a major impact on their physio-chemical characteristics. Because of their distinctive physical and chemical characteristics, NPs have found remarkable success in a wide range of applications in a variety of industries, including medicine, the environment, energy-based research, imaging, chemical & biological sensing, gas sensing, etc. Researchers have a stronger preference towards nanotechology[9].

COPPER OXIDE NANOPARTICLES

Copper oxide (CuO), a p-type semiconductor with a small indirect band gap of about 1.2 eV, has been acknowledged as an important industrial material for several useful applications, including catalysis, batteries, magnetic storage media, antibacterial composites, solar energy conversion, gas sensing, and field emission devices[10]. CuO is one of the best materials for electrical, optical, sensing, and other applications due to the possibility of low-cost manufacturing processes and the material's favorable electrochemical characteristics[11].

Copper oxide nanoparticles have stable chemical and physical characteristics, are relatively inexpensive, and are photocatalytic. CuO-NPs have the potential to be used as anti-infective agents due to their extraordinarily large surface area and attractive crystal morphologies[12]. CuO-NPs have a brownish-black color and are members of the monoclinic structural system. CuO nanoparticles (CuO-NPs) can be produced using a number of different physical and chemical techniques[13].

Method of preparation of CuO nanoparticles

CuO-NPs have been made using a variety of techniques, with varying shape and sizes. There are different physical, chemical and biological methods used for the preparation.

Physical method

Evaporation,condensation and laser ablation are the most popular physical synthesis methods utilized to produce metal nanoparticles. A tube furnace and atmospheric pressure are required for a physical technique. The absence of solvent contamination and the homogeneity of nanoparticle distribution are two benefits of a physical method over a chemical one. However, there are drawbacks to physical synthesis, including the size of the tube furnace, the high energy consumption, and the length of time required to obtain thermal stability (several tens of minutes to reach a stable operating temperature). Due to the temperature disparity between the area around the heater surface and the tube furnace, small nanoparticles are created by cooling evaporated vapors. Below is a list of a few of them.

Physical vapour deposition (PVD) : This technique includes depositing copper oxide nanoparticles from a vapor phase onto a substrate using techniques including sputtering, evaporation, or molecular beam epitaxy.

Chemical vapour deposition (CVD) : In this process, copper oxide nanoparticles are created by the breakdown of a precursor gas, often a metal organic molecule, in the presence of a heated substrate.

Laser Ablation: In this technique, a high-energy laser is used to create nanoparticles by abrading a target material, usually copper, in a liquid medium.

Ball milling: In this technique, copper oxide powder is ground to create nanoparticles in the presence of a milling agent.

Sol gel : The process involves dissolving a copper salt in a solvent, then adding a base to start condensation and hydrolysis reactions that produce nanoparticles.

Chemical method

In chemical synthesis, a product is created when a precursor is dissolved into a liquid (or solution) phase, forming a precipitate. Chemical synthesis is in opposition to top-down and gas-phase synthesis methods used in mechanical and physical methods of producing nanomaterials . The terms "wet synthesis methods" and "solution-phase synthesis" are also used to describe chemical synthesis techniques. During this method, the soluble components become insoluble or precipitate after becoming insoluble[14].

Some of the chemical methods are below

Solvothermal method : This approach is comparable to the hydrothermal approach, but a solvent is added to the mixture during the reaction to improve the size and shape and also to enhance the solubility of the reactants.

Hydrothermal method : This technique involves dissolving a copper salt in a solvent, which is then heated at high pressures and temperatures inside a container that is tightly sealed. CuO nanoparticles are produced as a result of this.

Reduction method : In this process, copper oxide nanoparticles are created by reducing a copper salt with a reducing agent such sodium borohydride or hydrazine.

Precipitation method : In this procedure, a copper salt is dissolved in water, and then copper hydroxide is produced by adding a precipitating agent, such as sodium hydroxide or ammonia. After that, heat is used to create copper oxide nanoparticles from the copper hydroxide.

Biological method

Biological copper NPs are synthesis , By using the reducing agent hydrazine and the stabilizer Psidiumguajava leaf extract in a microwave-assisted one-pot process. The leaf extract, hydrazine hydrate, and stock solution of CuSO4 5H2O are combined, and the mixture is thoroughly agitated for 2 minutes while maintaining a pH of 5. A vivid yellow color appears, and the solution is then exposed to 720 W of microwave radiation. The formation of CuNps are indicated by the color shift from orange to brickred [15].

Using hairpin DNA as a template, combined with a poly T loop and a random double-strand stem, we can create fluorescent copper nanoparticles. By using environmentally friendly synthesis techniques, it is possible to create copper nanoparticles in a single step. This is accomplished by soaking Impatiens balsamina leaf extract in a copper sulfate solution and periodically monitoring the absorption spectra. The development of copper nanoparticles is indicated by the presence of light red color.It is possible to create copper nanoparticles by separating the endophytic actinomycetes from the seaweeds.

Application of CuO NPs

Antimicrobial drugs are the major application for CuO nanoparticles. They are employed in hospitals because , they have an antibacterial ability to kill more than 99.9% of Gram-positive and negative bacteria within two hours of exposure[16]. CuO may be used against nosocomial infections as a disinfectant. Due to their potent bactericidal properties against various Gram + ve and Gram -ve bacterial strains, they are used in wound dressings. Copper deficiency diseases such cardiovascular problems, anemia, and osteoporosis are treated with the help of biosynthesized copper nanoparticles in combination with already-available medications. They are also utilized as a cofactor for numerous enzymes, dietary supplement, biosensor, antibacterial agent, and in the treatment of cancer. Because of their improved photocatalytic properties, the copper nanoparticles made from *I. balsamina* using green technology can be used to degrade harmful organic dyes. Additionally, they are used to detect dangerous mercury ions up to 1 ppm in concentration.It has been proven that actinomycetes helped in the manufacture of copper nanoparticles, and these particles showed improved control over human pathogenic germs., Since copper nanoparticles serve as a container for electron exchange with the microbe, they have a stronger antibacterial and inhibitory effect on pathogens including *Staphylococcus saprophyticus*, *Pseudomonas aeruginosa*, and *Staphylococcus epidermidis*.

The antibacterial properties of the copper nanoparticles made from *Syzygiumaromaticum* bud extract are demonstrated by their zone of inhibition at 8 mm and 6 mm, respectively, against *Bacillus* spp. and *Penicillium* spp

The water-soluble, synthetic nanoparticles have been discovered to be highly effective against fungi and to exhibit long-term durability.In comparison to silver nanoparticles and other antibiotics, the CuNPs have demonstrated superior antibacterial activity. They are also employed in DNA hybridization.These copper nanoparticles are used in alkyne and azide as catalysts.By serving as a fluorescence probe, the copper nanoparticles are used to detect the presence of cyanide ions in aqueous solution. The metallic copper nanoparticles have stronger catalytic activity than commercial powder, and are therefore utilized to speed up the Ullmann's reaction as well as to create binary alloys of metallic clusters.

Copper nanoparticles are a class of materials with unique features that are used in a variety of applications, including magnetic, food, electronic, biological, pharmacological, drug, cosmetic, energy, catalytic, and materials.

HYDROTHERMAL SYNTHESIS OF NANOPARTICLES

One of the most frequent ways for preparing nanomaterials is hydrothermal synthesis. It is essentially a solution-reaction technique. The creation of nanomaterials in hydrothermal synthesis can occur at temperatures ranging from ambient temperature to extremely high temperatures. Depending on the vapor pressure of the main composition in the reaction, either low-pressure or high-pressure conditions can be utilized to control the morphology of the materials to be synthesized. Using this method, many different types of nanomaterials have been successfully produced. The hydrothermal synthesis method has major advantages over other methods. Hydrothermal synthesis can produce nanomaterials that are unstable at high temperatures. The hydrothermal process can generate nanomaterials with high vapor pressures with minimal material loss[a].

Crystal synthesis or crystal development under high presure and high temperature, water conditions from compounds that are insoluble at ordinary temperature and pressure (100 °C, 1 atm) is commonly referred to as hydrothermal synthesis[c]. The term "hydrothermal" derives from geology[b].

In the hydrothermal method ,the synthesis of the nanoparticleis depend on the solubility of the species in the reaction in a medium of water at a desired pressure. An ‘autoclave’is a stainless steel chamber with a Teflon coating that is used in the hydrothermal process .The pre-synthesized chemical species are moved to Teflon-lined autoclave, where the nutrient is supplied with water. The autoclave is kept at a specific temperature, creating an internal pressures which stimulates crystal formation and dissolves the nutrient[d].

Autoclave and Teflon vessel :

Advantage of hydrothermal process

- ✓ This technique enables the crystallization of substances with high melting points at reduced temperatures.
- ✓ Using the hydrothermal process, materials with high vapour pressure close to their melting temperatures can be created.
- ✓ The process is also very effective for growing massive, high-quality crystals while preserving compositional control.

Disadvantage of hydrothermal process

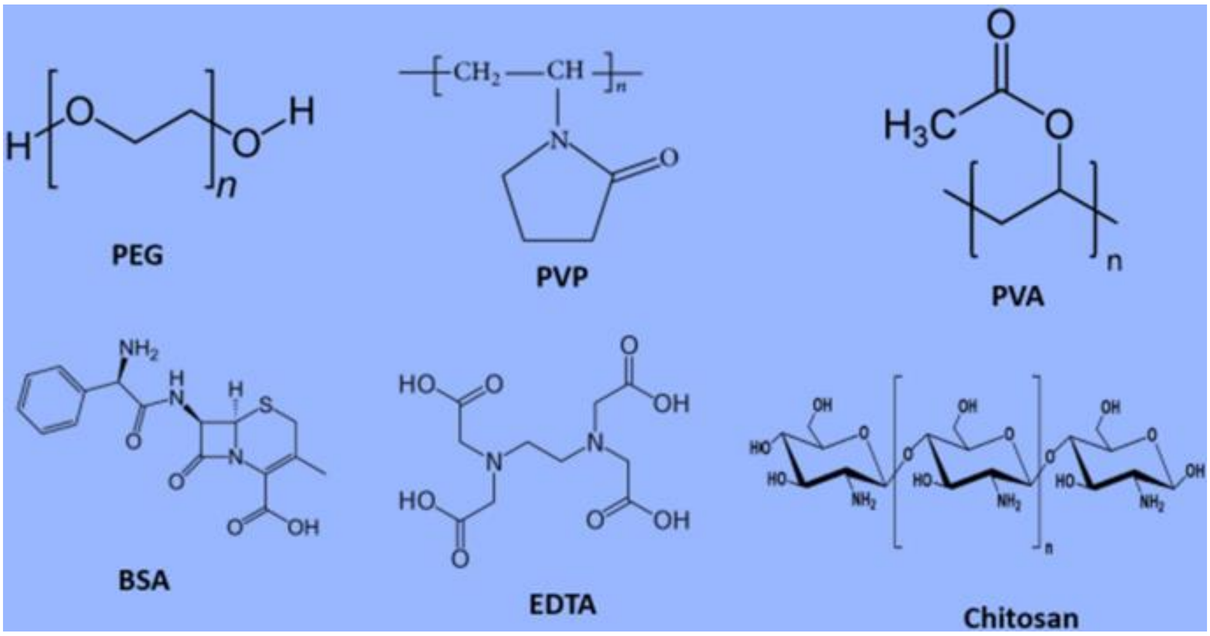
- ✓ The requirement for expensive autoclaves
- ✓ It is difficult to watch the crystal grow.

CAPPING AGENT IN NANOPARTICLE SYNTHESIS

Researchers are particularly interested in evaluating nanoparticles' potential for therapeutic treatment and their impact on the environment. Surface capping improves the biological characteristics of nanoparticles. capping agents exhibit clinical value in concert with the biocompatible nanoparticles to which they have been bonded. The steric barrier caused by the covalent interaction between the chains of capping ligands and the surface of the nanoparticles gives the nanocomposite its ultimate stability. At the nanoscale, there are more atoms on the surface, and capping boosts this number even more. Additionally, by using the proper capping agents, the aggregation of nanoparticles is reduced for a longer period of time[w].

The capping agent is defined as an amphiphilic molecule with a non-polar hydrocarbon tail and a polar head group. Capping agents confer functionality and improve compatibility with the another phase, because of their amphiphilic character. While the polar head interacts with the metal atom of the nanosystem, the non-polar tail interacts with the surrounding medium. Various capping agents, such as tiny ligands,surfactants, cyclodextrins,dendrimers , polymers, and polysaccharides, have been utilized in the creation of nanoparticles. Each of these has been effectively used as a capping agent, causing modest modifications in nanoparticles that reveal a powerful medicinal and environmental cleansing impact.

Some of the commonly used capping agents : Polyethylene glycol (PEG),polyvinyl alcohol (PVA),polyvinylpyrrolidone (PVP), bovine serum albumin (BSA),ethylene diamine tetra acetic acid (EDTA)



POLYETHYLENE GYLCOL (PEG)

PEG, which has the structural formula H-(O-CH2-CH2)n-OH, is an excellent biocompatible polymer. Ethylene glycol is poly-condensed during the process of making PEG in the presence of an acidic or basic catalyst, resulting in a product with a lower molecular weight. Polyethylene glycol is soluble in both organic and aqueous solutions. This feature improves both its processability and biocompatability. It is non-immunogenic and less harmful. As a result, it is used as a surface modification with other substances such as micelles, particles and biomaterials for the transportation of active molecules as well as for the creation of physical and chemical hydrogels. PEG does not hydrolyze into a detached state in vivo. However, the polymer has a higher biodegradability and water affinity due to its hydrophilic characteristics

PEG has been widely employed as a capping agent for a variety of applications, including the biomedical.

CHARACTERIZATION TECHNIQUES

There are various technique used for the characterization of PEG capped CuO nanoparticles ,these comprise of powder Xray diffraction , ultraviolet visible spectroscopy , transmission electron microscopy (TEM), wide angle x ray diffraction (WAXD), electron spin resonance, fourier transform infrared spectroscopy (FTIR), x ray photoelectron spectroscopy (XPS), laser particle analyzer. Using this, information about the sample is obtained. The technique used for the characterization of PEG capped CuO NPs for this work are FESEM,FTIR,UV,XRD

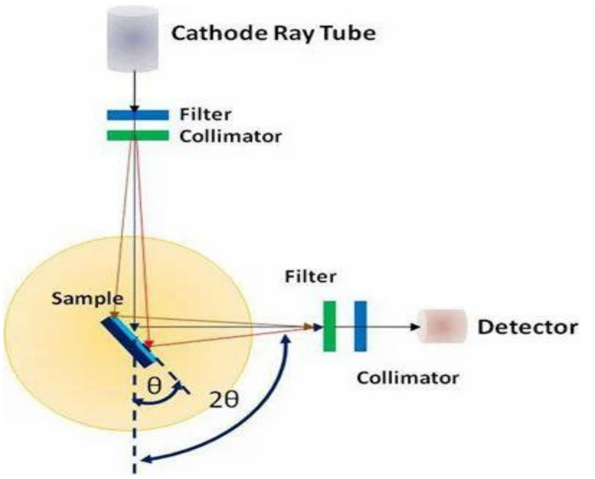
1**, field emission scanning electron microscopy (FESEM)**

Field emission scanning electron microscopy (FE-SEM) is a sophisticated technique for capturing the microstructure image of materials. It provides elemental information about the object at magnifications ranging from 10x to 300,000x and with a practically limitless depth of field. It produces simpler, less electrostatically deformed images.

FESEM apparatus

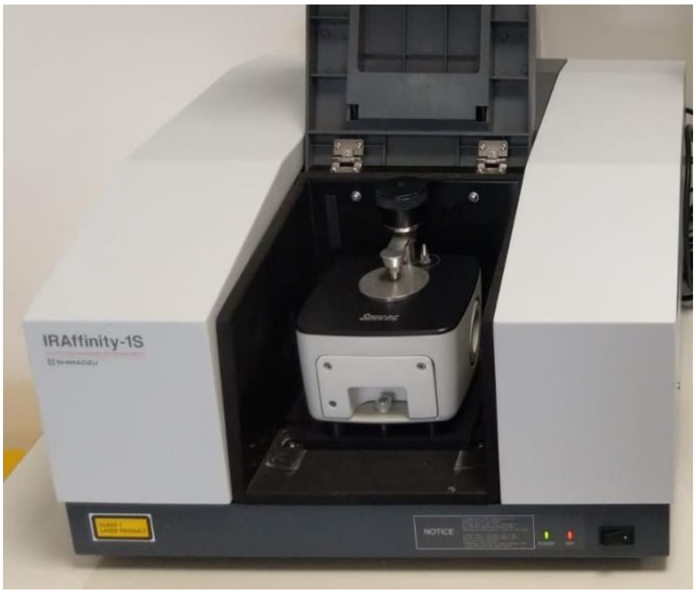
2**, X-Ray diffraction (XRD)**

X-Ray diffraction (XRD) is a nondestructive method for determining a material's chemical composition, physical attributes and crystallographic structure . It works by using the constructive interference of monochromatic X-rays with a crystalline sample[14]. In this procedure, a powdered sample is subjected to an X-ray beam, which diffracts off the atoms in the material's crystal lattice. The diffracted X-rays form a diffraction pattern that is unique to the sample's crystal structure.



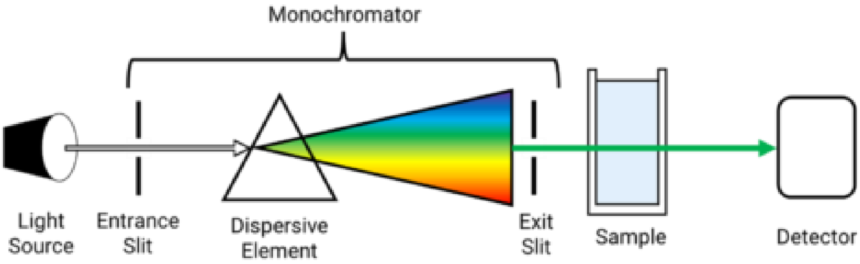
3, Fourier transform infrared spectroscopy (FTIR)

Fourier-transform infrared spectroscopy (FTIR) is a technique for obtaining emission spectra and absorption spectra of a liquid, gas, or solid.The phrase Fourier-transform infrared spectroscopy is used because it need a mathemactical process called Fourier transform to turn raw data into the real spectrum.Absorption spectroscopy techniques are used to determine how much light a material absorbs at each wavelength[15].



4, ultraviolet spectroscopy (UV)

UV-Visible Principle Spectroscopy is based on the absorption of visible or ultraviolet light by chemical substances, which leads in the formation of different spectra.The Spectroscopy is based on the interaction of matter and light. The formation of spectrum is because of the excitation and the de-excitation of the matter when it absorb the light..



CHAPTER 2

SCOPE AND OBJECTIVES

Due to the unique physical and chemical features of the Nanomaterials, it become an indispensable tool in modern science and technology. The metal oxide Nanoparticles such as copper oxide Nanoparticles have wide variety of application in biomedical field. The copper oxide nanoaparticles have effective biological activity such as Antimicrobial ,Antioxidant, Anticancer and cytotoxic activity.

The purpose of this work is to synthesize Hydrothermally produced PEG capped copper oxide Nanoparticles

OBJECTIVES

- Synthesis of PEG capped Copper Oxide Nanoparticles
- Hydrothermal technique employed for the synthesis
- Characterization of PEG capped copper oxide Nanoparticles using IR,UV,XRD,and FESEM

CHAPTER 3
EXPERIMENTAL PART

MATERIALS REQUIRED

- ✓ Copper Acetate Monohydrate ($C_4H_8CuO_5$)
- ✓ Glacial Acetic Acid
- ✓ Sodium Hydroxide (NaOH pellets)
- ✓ Polyethylene glycol 6000
- ✓ Distilled Water

PROCEDURE

Synthesis of PEG Capped Copper oxide Nanoparticles Using Hydrothermal Method

- A 10mm solution of Copper Acetate Monohydrate is added to 1% Glacial Acetic Acid, which contains 0.05% polyethylene glycol 6000 (1 ml of Glacial Acetic Acid in 99 ml of distilled water).The mixture is then swirled magnetically for 24 hours at a speed of 420 to 450 RPM.

- Then pH was then maintained at 12 by adding 6 mM NaOH solution and agitating it magnetically for 30 minutes.

- Afterward, approximately 75 ml of this solution is put into a Teflon-lined autoclave and heated to 100 °C for seven hours. The raw materials were then separated by centrifugal filtration, washed in distilled water till the pH attained 7, and dried for 24 hours in an oven at 100 °C.The resulted nanoparticles are then charcterised by FTIR,XRD,UV and FESEM.

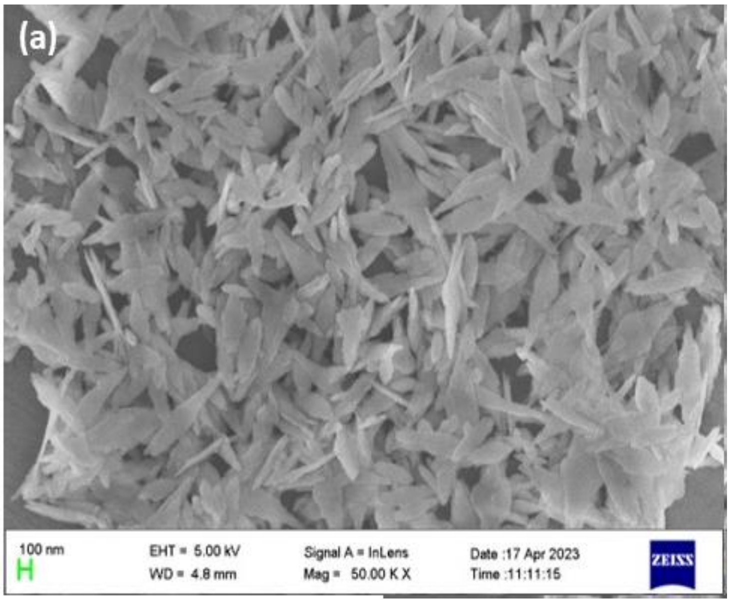
CHAPTER 4

RESULT AND DISCUSSION

CHARACERTIZATION OF PEG CAPPED COPPER OXIDE NANOPARTICLES

FESEM ANALYSIS

The figure below shows the FESEM surface morphological image of PEG-capped copper oxide.



It is evident from the depiction that the morphology of the PEG-capped Cuo nanoparticles be like grains.

X RAY DIFFRACTION ANALYSIS

For PEG-capped CuO, the average crystallite size is 16.6 nm**.**

Figure 1 displays the sample's powder XRD pattern. All of the diffraction peaks for CuO are the result of the monoclinic structure of PEG-capped CuO with 2 theta values and the associated diffraction planes 35.7(002), 38.9(111), 48.7(202), 58.3(202), 61.7(113), 66.3(311), and 68.3(220).

CuO has solid XRd peaks, which suggests that these metal oxides are naturally crystalline.Thus, the XRD patterns proved that the provided CuO was a pure metal oxide with no additional impurities . The CuO NPs is phase pure as well

The dedye-scherrer formula has been used to determine the average metal oxide crystal size.

$$D = k\,\lambda/\cos\theta\beta$$

Where D is the crystallite size , K is a dimensionless constant and it may vary from 0.89 to 1.39 depending on the precise geometry of the scattering substances (here it was taken as 0.94) , λ is the wavelength of X ray (1.5406 Å for Cu Kα Radiation), β is full width at half maximum of the XRD peak, and Ɵ is the diffraction angle and it is obtained from the 2Ɵ value of the peak with maximum diffraction intensity in the XRD patten.

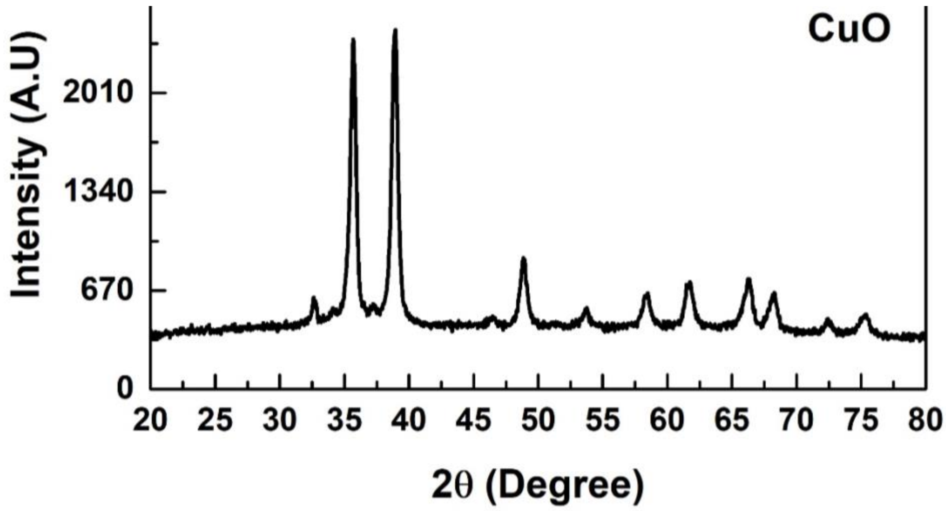


Figure 1 : The PEG-capped CuO's XRD peak pattern

CHAPTER 5

CONCLUSION

CuO nanoparticles were created using a hydrothermal technique in a stainless steel autoclave ,heated to 100 °C.Polyethylene glycol was used as a capping agent.The nanoparticles were discovered to have a grainy texture.The phase structure was validated by XRD examination and possesses a monoclinic phase, as well as a particle size of (16.6nm).

CHAPTER – 6

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