BHARATA MATA COLLEGE, THRIKKAKARA

Re-accredited by NAAC with A+ Grade

DEPARTMENT OF PHYSICS

MAHATMA GANDHI UNIVERSITY

2020-2023

A PROJECT REPORT ON

SYNTHESIS AND STRUCTURAL CHARACTERISATION OF CuO NANOPARTICLES

PROJECT SUBMITTED BY:

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CERTIFICATE

This is to certify that **DEVANG VIJAY, Reg No: 200021039167** final year student of **BSc. PHYSICS MODEL 2** of this institute has done this project, during this academic year **2020-2023**.

Project Guide: **Dr. ANU PHILIP**

Date:

Internal Examiner:

Head of department: **Dr. ANU PHILIP**

Date:

External Examiner:

Date:

ACKNOWLEDGEMENT

I would like to express my sincere gratitude to all my teachers in the **DEPARTMENT OF PHYSICS, BHARATA MATA COLLEGE, THRIKKAKARA.**

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I would also like to thank my project partner **GIL JHONSON** for his support and cooperation throughout the work.

CONTENT

INTRODUCTION

1. NANOTECHNOLOGY

Nanotechnology is a branch of science and technology that deals with the design, creation, manipulation, and application of materials, devices, and systems at the nanoscale, which is typically considered to be between 1 to 100 nanometers (nm) in size.

At the nanoscale, the properties of materials can differ significantly from their bulk counterparts, and new and unique properties can emerge. Nanotechnology involves the use of tools and techniques to manipulate matter at the atomic and molecular level, allowing scientists to create new materials and devices with unprecedented precision and control.

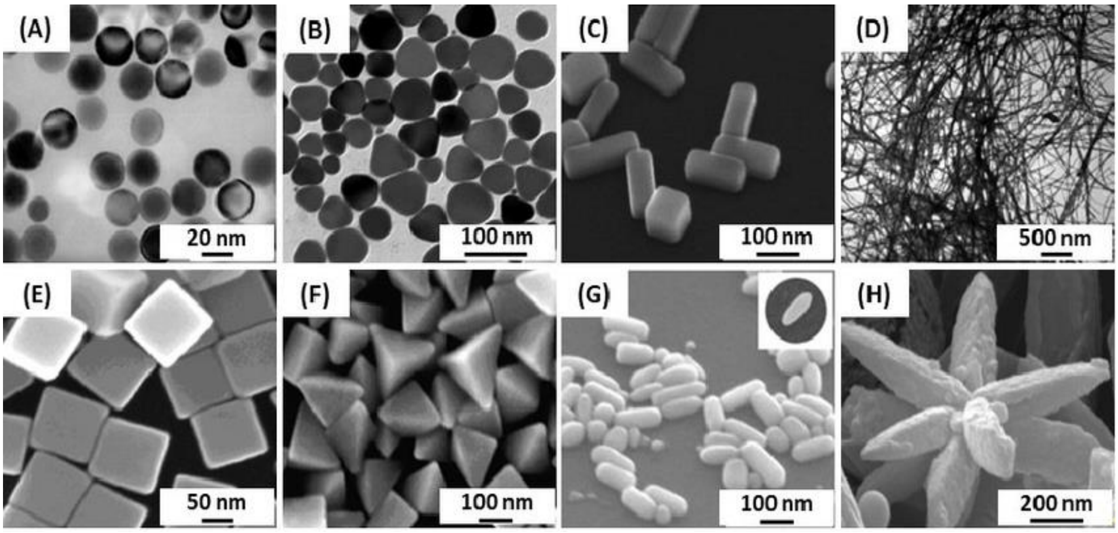
Some of the applications of nanotechnology include the development of new drug delivery systems, more efficient energy sources, smaller and faster electronic devices, and stronger and lighter materials. Nanotechnology has the potential to revolutionize many areas of science and technology, and its continued development is an active area of research and innovation.

2. NANOPARTICLES

Nanoparticles are tiny particles with a size range between 1 and 100 nanometers, which is typically much smaller than the size of human cells. These particles can be made from a variety of materials such as metals, semiconductors, polymers, and ceramics. Due to their small size and unique properties, nanoparticles have many potential applications in fields such as medicine, electronics, energy, and materials science.

Nanoparticles can be engineered to have specific properties such as size, shape, surface area, and reactivity, which can make them useful for a wide range of applications. For example, in medicine, nanoparticles can be used to deliver drugs to specific cells or tissues in the body, while in electronics, they can be used to make smaller and more efficient electronic devices.

However, the small size and high surface area of nanoparticles can also make them potentially hazardous to human health and the environment, as they can penetrate biological barriers such as cell membranes and accumulate in tissues. Therefore, the safe use and disposal of nanoparticles is an important consideration in their development and application.



3. SYNTHESIS

There are various methods for synthesizing nanoparticles, which can be broadly categorized into top-down and bottom-up approaches. Here are some common types of nanoparticle synthesis methods:

1. Chemical Reduction: This method involves reducing metal ions in a solution using a reducing agent, resulting in the formation of nanoparticles. The particle size and shape can be controlled by varying the reaction conditions and the concentration of the reactants.

2. Sol-Gel Synthesis: This method involves the hydrolysis and condensation of metal alkoxides in a liquid medium to form a sol, which is then dried and heated to form nanoparticles. This method can produce highly pure nanoparticles with good control over the size and shape.

3. Hydrothermal Synthesis: This method involves the reaction of metal salts with a solution under high temperature and pressure conditions in an autoclave. This method can produce highly crystalline nanoparticles with a narrow size distribution.

4. Microemulsion Synthesis: This method involves the formation of microemulsions, which are tiny droplets of one liquid suspended in another, to create a confined reaction environment for the synthesis of nanoparticles. This method can produce nanoparticles with a narrow size distribution and good stability.

5. Green Synthesis: This method involves the use of natural compounds such as plant extracts or microorganisms to synthesize nanoparticles, without the use of toxic chemicals or solvents. This method is eco-friendly and can produce nanoparticles with unique properties.

6. Physical Vapor Deposition: This method involves the deposition of a thin film of metal onto a substrate by physical vaporization of the metal using techniques such as sputtering or evaporation. The film can then be broken down into nanoparticles by annealing or etching. This method can produce nanoparticles with precise control over size and shape.

These are just a few examples of the many methods for synthesizing nanoparticles, and each method has its advantages and disadvantages depending on the desired properties of the nanoparticles and the intended application.

4. CHARACTERISATION

Characterization of nanoparticles is an important step in understanding their properties and behavior, as well as in determining their suitability for various applications. Some of the common methods for characterizing nanoparticles are:

Size and morphology analysis: Techniques such as transmission electron microscopy (TEM), scanning electron microscopy (SEM), and atomic force microscopy (AFM) can be used to visualize and measure the size and shape of nanoparticles.

Composition analysis: Techniques such as X-ray diffraction (XRD) and energy-dispersive X-ray spectroscopy (EDX) can be used to determine the elemental composition of nanoparticles, while techniques such as Fourier transform infrared spectroscopy (FTIR) and Raman spectroscopy can be used to identify the chemical bonds present in the particles.

Surface area and charge analysis: Techniques such as Brunauer–Emmett–Teller (BET) analysis and zeta potential measurements can be used to determine the surface area and charge of nanoparticles.

Thermal analysis: Techniques such as differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) can be used to study the thermal behavior of nanoparticles, including their melting point, decomposition temperature, and stability.

Biological analysis: Techniques such as in vitro and in vivo toxicity assays can be used to evaluate the potential toxicity of nanoparticles and their effects on biological systems.

Overall, a combination of these and other techniques can be used to fully characterize the properties of nanoparticles and to understand how they might behave in different environments and applications.

5. APPLICATIONS

Nanoparticles have a wide range of potential applications in various fields, including medicine, electronics, energy, and materials science. Here are some of the common applications of nanoparticles:

Drug delivery: Nanoparticles can be used as carriers for drugs, allowing for targeted and controlled drug delivery to specific cells or tissues in the body. They can also enhance the solubility and stability of drugs and improve their pharmacokinetics.

Imaging and diagnostics: Nanoparticles can be used as contrast agents for medical imaging, such as magnetic resonance imaging (MRI), computed tomography (CT), and ultrasound. They can also be used as biosensors for detecting disease markers and other biomolecules.

Environmental remediation: Nanoparticles can be used to remove pollutants from soil and water, and to degrade organic contaminants through photocatalysis or other mechanisms.

Energy: Nanoparticles can be used to improve the efficiency and performance of energy storage devices, such as batteries and supercapacitors, as well as in solar cells and fuel cells.

Catalysis: Nanoparticles can be used as catalysts for chemical reactions, such as in the production of fuels, chemicals, and pharmaceuticals.

Electronics: Nanoparticles can be used to make smaller and more efficient electronic devices, such as transistors, memory devices, and displays.

Coatings and surfaces: Nanoparticles can be incorporated into coatings and surfaces to improve their properties, such as scratch resistance, wear resistance, and antimicrobial activity.

These are just a few examples of the potential applications of nanoparticles, and research in this area is ongoing, with many new and exciting applications being explored.

MATERIALS AND METHODS

COPPER OXIDE (NANOPARTICLES)

Copper oxide nanoparticles are nanoparticles made of copper and oxygen atoms, with a particle size between 1 and 100 nanometers. They can exist in different forms, such as cupric oxide (CuO) and cuprous oxide (Cu_2O), depending on the oxidation state of the copper atoms.

Copper oxide nanoparticles have many potential applications due to their unique properties, such as high surface area, optical and electrical properties, and catalytic activity. They can be used as catalysts in various chemical reactions, as antimicrobial agents, in gas sensors, and in solar cells, among other applications.

Copper oxide nanoparticles have also been studied for their potential toxicity and environmental impact, as they can accumulate in living organisms and have been shown to cause oxidative stress and DNA damage. Therefore, the safe use and disposal of copper oxide nanoparticles is an important consideration in their development and application.

APPLICATIONS

Copper oxide nanoparticles have a wide range of potential applications due to their unique properties, such as high surface area, optical and electrical properties, and catalytic activity. Here are some of the common applications of copper oxide nanoparticles:

Catalysis: Copper oxide nanoparticles can be used as catalysts in various chemical reactions, such as in the production of methanol, ethanol, and other chemicals. They can also be used as photocatalysts for water splitting and other energy conversion processes.

Antimicrobial agents: Copper oxide nanoparticles have been shown to have antimicrobial properties, and can be used as coatings on medical devices and in textiles to prevent the spread of infections.

Gas sensors: Copper oxide nanoparticles can be used as gas sensors to detect and measure the concentration of various gases, such as carbon monoxide and nitrogen oxides.

Solar cells: Copper oxide nanoparticles can be used in the development of solar cells, as they can absorb light in the visible and near-infrared regions of the spectrum and have the potential to improve the efficiency of solar energy conversion.

Batteries and supercapacitors: Copper oxide nanoparticles can be used in the development of high-performance batteries and supercapacitors, as they have high electrical conductivity and can store and release energy efficiently.

Water treatment: Copper oxide nanoparticles can be used to remove pollutants from water, such as heavy metals and organic compounds, through adsorption and catalytic degradation.

These are just a few examples of the potential applications of copper oxide nanoparticles, and research in this area is ongoing, with many new and exciting applications being explored.

METHOD OF PREPARATION

Hydrothermal Synthesis

Hydrothermal synthesis is a method for creating inorganic compounds, such as nanoparticles and crystals, by using high-pressure and high-temperature water or aqueous solutions as the reaction medium.

In this process, a precursor material is dissolved in water or a solvent and sealed in a reaction vessel. The vessel is then heated to a high temperature and pressure, typically above 100°C and several hundred atmospheres, and allowed to react for a period of time. The high pressure and temperature conditions can lead to the formation of new compounds and the growth of crystals or nanoparticles.

The hydrothermal synthesis method has several advantages, including the ability to create high-quality materials with controlled size, shape, and composition, as well as the ability to synthesize materials that are difficult or impossible to obtain by other methods. It is used in a wide range of fields, including materials science, nanotechnology, and environmental science.

EXPERIMENTAL

1. APPARATUS OF THE PROJECT

- Beaker
- Watch Glass
- Measuring Jar
- Teflon autoclave
- Stirrer
- Laboratory oven
- Spatula

- Funnel
- Filter Paper

CHEMICLAS USED

- Copper Malachite [$CuCo_3Cu(OH)_2$]
- Ammonium Hydroxide [NH_4OH]
- Distilled water
- Polyethylene glycol (PEG)

Calculation of mass of Copper Malachite [$CuCo_3Cu(OH)_2$]

- Required volume (V) = 50 ml

- Molecular mass of $CuCo_3Cu(OH)_2$ (m) = 221.116 g/mol

- Required molarity of the solution (M) = 5×10^{-3} mol/L

- Mass of $CuCo_3Cu(OH)_2$ (X) = { m x M x V} / 1000

= {221.116 x 5 x 10^{-3} x 50} / 1000

= 0.055279 g

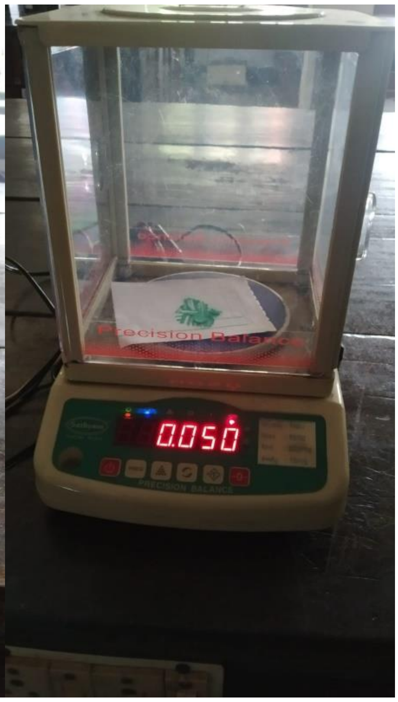
∴ The required mass of $CuCo_3Cu(OH)_2$ is 0.055279 g.

SYNTHESIS STEPS

0.055279GRAMS of $CuCo_3Cu(OH)_2$ is dissolved in distilled water to prepare 50ml solution of 0.005 molarity. Then it is mixed with 0.1 gram of PEG and 50ml distilled water. The solution is stirred for 30 minutes. Then 5ml of NH_4OH is added to the solution. Then the solution is again stirred for 2 hours so that it turns from aqua blue to intense blue. Then the solution is transferred into a Teflon autoclave and placed in a domestic oven. Then it is heated upto 200^0c to study the synthesis of CuO nanoparticles at temperature. Then the resultant CuO powder is washed with distilled water at dried at 70^0c in a hot plate.


MAINS ON
HEATER ON
PV
220
SP
225
Honeywell
DC1040
DTC


REMI 2MLH
0540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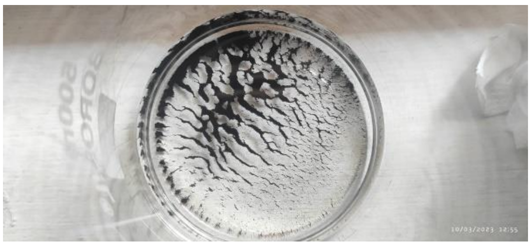


1 RESULTS AND DISCUSSIONS

XRD Result of CuO nanoparticles synthesized at 200^{0}c

Structural characterization using x- ray diffraction

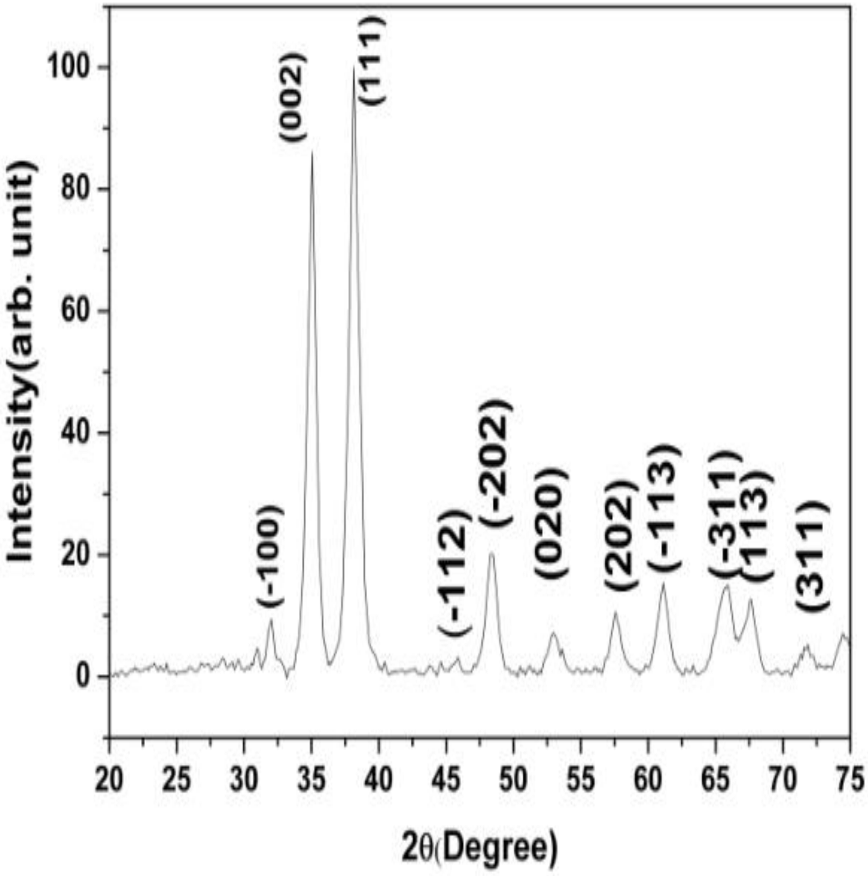


Figure 1 :XRD pattern for the prepared CuO nanopowder.

Figure1 shows the XRD pattern obtained for the prepared CuO nanoparticles. All the major diffraction peaks are indexed and these values are consistent with "JCPDS" for monoclinic phase CuO. In the X– ray diffraction high intensity peaks located at 35 and 38.5 corresponds to reflection from (002) and (111) atomic planes respectively. X-ray diffraction peaks which corresponds to phases other than monoclinic phase was found absent. Lower intensity peaks observed at 32.5, 48.5, 57.5, 62, 65.5, 67.5, 73, 74.5

correspond to reflection from (-100), (-112), (-202), (020), (202), (-113), (-311),(113), (311) atomic planes.

The crystallite size (D) was calculated from peak broadening using the Scherrer approximation formula

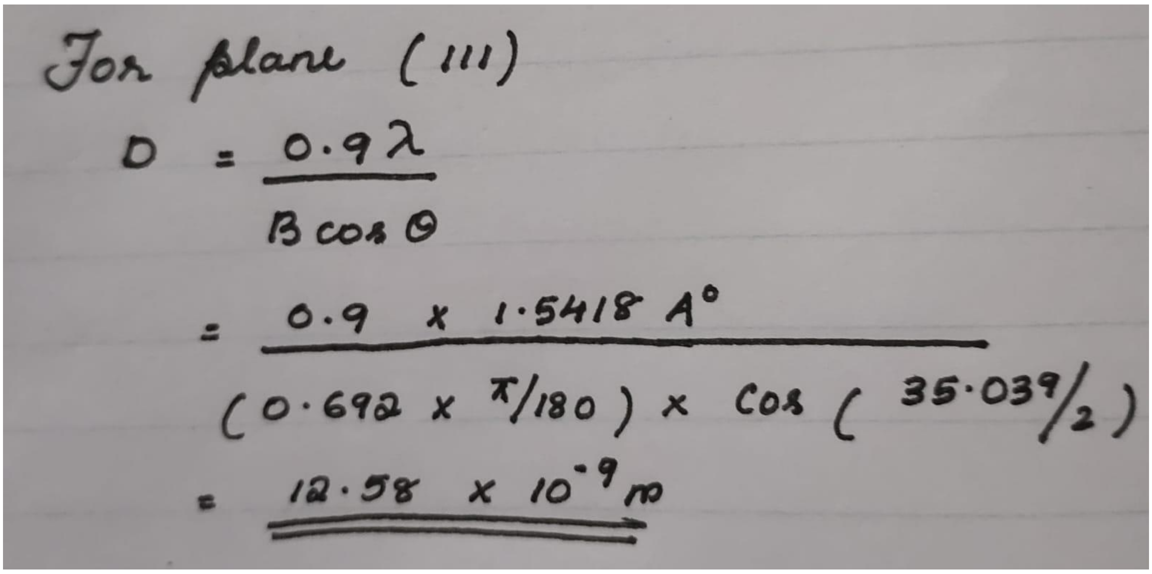
$$D = \frac{0.9\lambda}{B cos\theta},$$

Where λ is the wavelength of the X-ray (1.5418 Å),

B the full-width at half maximum. (FWHM,radian)

and θ the Bragg angle (degree).

The crystallite sizes were calculated using FWHM of the peaks corresponding to the planes (111), (0 0 2) and the average crystallite size was found to be 11.34 nm.



For plane (111)

$$D = \frac{0.9\lambda}{B \cos\theta}$$

$$= \frac{0.9 \times 1.5418\ A^{\circ}}{(0.692 \times \pi/180) \times \cos(35.039/2)}$$

$$= 12.58 \times 10^{-9}\ m$$

Surface morphological Analysis using scanning electron microscopy

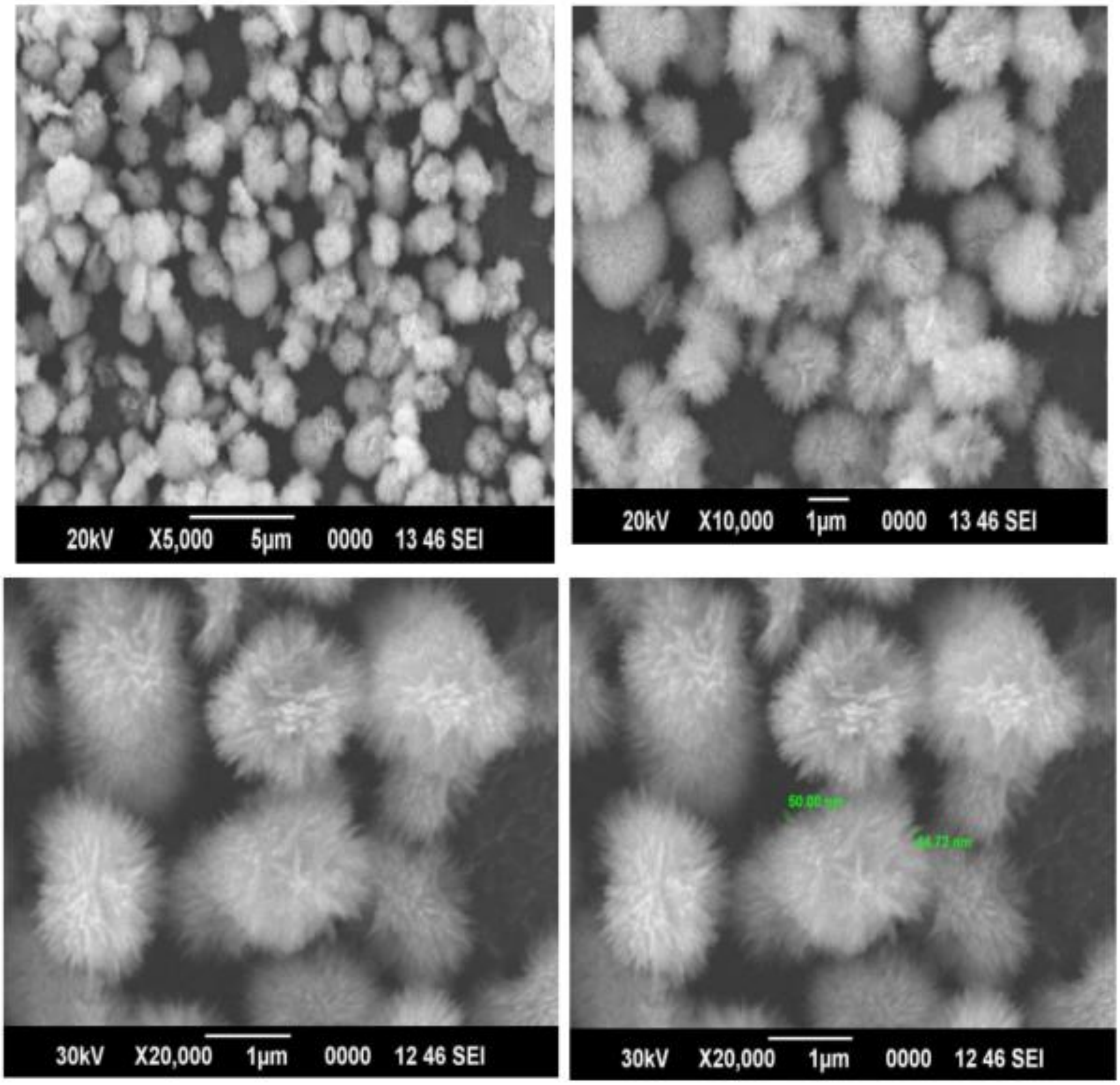


Figure2: SEM images of CuO nanoparticles with different magnifications.

Surface morphology of the prepared nanoparticles were analysed using SEM. Figure 2 shows the SEM images of CuO nanoparticles with different magnification. The SEM image shows a flower like structure with large surface to volume ratio. The needle likepetals surrounds the circular corestructure with the needle size around 40 – 50 nm. This is a structure fine enough for gas sensing applications.

CONCLUSION

Hydrothermal technique was used to prepare CuO nanoparticles.

X-ray Diffraction and SEM were used to structurally characterize the CuO nanoparticle.

Flower-like CuO nanostructures were confirmed using SEM.

The prepared CuO nanostructures can be used for NH_3 gas sensing.

Acknowledgement

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- https://ec.europa.eu/health/scientific_committees/opinions_layman/en/nanotechnologies/l-2/1-introduction.htm#:~:text=Nanotechnology%20refers%20to%20the%20branch,of%20a%20millimetre)%20or%20less.
- https://pubs.aip.org/aip/acp/article/2335/1/100001/1000750/Hydrothermally-synthesized-CuO-nanostructures-and
- https://instanano.com/all/characterization/xrd/crystallite-size/
  (For crystal size)