

**BHARATA MATA COLLEGE, THRIKKAKARA**

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**DEPARTMENT OF PHYSICS**

MAHATMA GANDHI UNIVERSITY

2020-2023



A PROJECT REPORT ON

**SYNTHESIS AND OPTICAL CHARACTERISATION**

**OF CuO NANOPARTICLES**

PROJECT SUBMITTED BY:

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## **CERTIFICATE**

This is to certify that **ANJALI BINU**, Reg No: **200021039161** 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:

Date:

External Examiner:

Date:

## **ACKNOWLEDGEMENT**

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# INTRODUCTION

## NANOTECHNOLOGY

Since the turn of the century, researchers have been studying nanotechnology. Since Nobel laureate Richard P. Feynman presented the idea of "nanotechnology" in his notable 1959 talk "There's Plenty of Room at the Bottom" (Feynman, 1960), the area of nanotechnology has seen a few advancement headways.

## NANOPARTICLES

A nanoparticle is a small particle that ranges in size from one to one hundred nanometers. The physical and chemical properties of nanoparticles, which are undetectable to the naked eye. Nanoparticles are distinguished from microparticles (1-1000 m), "fine particles" (sized between 100 and 2500 nm), and "coarse particles" (sized between 2500 and 10,000 nm) by their smaller size, which influences very different physical or chemical properties such as colloidal properties, ultrafast optical effects, or electric properties.

Because of their unique material qualities and submicroscopic size, manufactured nanoparticles may be useful in a variety of sectors, including medicine, engineering, catalysis, and environmental cleanup.

## PROPERTIES

At the point when researchers found that a substance's size could influence its physicochemical characteristics, for example, its optical capacities, they understood the meaning of these materials. The ordinary shades of 20-nm gold (Au), platinum (Pt), silver (Ag), and palladium (Pd) NPs are, individually, wine red, yellowish dim, dark, and dim dark.. These NPs demonstrated distinguishing hues and qualities along with variations in size and shape that can be used in bioimaging applications. Aspect ratio, nanoshell thickness, and gold concentration variations all affect the solution's colour. A change in any of the factors mentioned above affects the NPs' ability to absorb light, leading to the observation of various absorption colours.

# SYNTHESIS

The phrase "nanoparticle synthesis" refers to the methods used to create nanoparticles. Nanoparticles can be produced through "bottom-up" processes, such as nucleating and growing particles from tiny molecular distributions in liquid or vapour phase, or they can be produced from larger molecules. Another method of synthesis is functionalization by conjugation to bioactive compounds. High yield and inexpensive nanomaterial synthesis has been a significant problem ever since the very early stages of the development of nanoscience.

# CHARACTERIZATION

When compared to the same bulk material, nanomaterials' dynamic optical properties, especially absorption, transmission, reflection, and light emission, might change greatly. By adjusting its form, size, and surface functioning, a variety of optical effects may be created for a number of purposes. Depending on the composition, size, and orientation, several methods may be used to accomplish this alteration (Smith and Nie, 2010). There are several ways in which a nanomaterial's optical quality is crucial. The alteration in their form, size, or kind may have an impact on the colour they create, since they are capable of constricting their electrical characteristics to cause quantum effects.

Optical Properties.

1. Colour and transparency are regarded as examples of optical characteristics. At the nanoscale, these qualities are shown to vary. For instance, gold appears yellow in bulk while appearing red in nanoscale.
2. Nanosized silicon appears red, but bulk silicon is grey in tone.
3. Zinc oxide, which in large quantities scatters visible light, inhibits ultraviolet light, and gives off a white look. While visible light's wavelength and the particle size of nanoscale zinc oxide are significantly different, the latter does not scatter the latter. As a result, it seems transparent.

Reason for change in optical properties in nanoscale:

Nanoparticles' tiny size limits the amount of freedom that their electrons can have. The main reason for changes in optical properties is how light waves travel in bulk matter. At the nanoscale level, characteristics. Light affects nanoparticles differently than it does larger ones.

# KEYS TO THE PROJECT

## CuO Nanoparticles

Despite the fact that they are not currently being extensively researched as an effective drug delivery vehicle, CuO nanoparticles offer exceptional biological features, including strong antibacterial activity against a variety of diseases as well as bacteria that are drug resistant. These qualities have inspired the development of a number of techniques with immediate biological applications, such as specially designed surfaces with antimicrobial properties, wound dressings, and modified fabrics. The development of intelligent systems for both pathogen detection and disease treatment is also thought to benefit from the use of these nano systems, which are considered to be potent alternatives.

The unique chemical and physical properties of nanomaterials, which differ from those of their bulk counterparts and include but are not limited to diffusivity, electrical resistivity, electrical conductivity, strength and hardness, chemical reactivity, and diverse and versatile biological activity, have spurred an exponential rise in research interest in them.

Metal oxide nanoparticles have attracted the most attention because they are widely used in industrial catalysts, chemical sensing devices, medical applications, disinfection, etc.

Metal oxide nanoparticles, such as copper oxide (CuO), have drawn interest primarily due to their antibacterial and biocidal capabilities and are potentially useful in numerous biological applications. It has been utilised in a variety of applications, including the creation of supercapacitors, near-infrared filters, magnetic storage media, sensors, catalysis, semiconductors, etc. Copper oxide is a semiconductor metal with special optical, electrical, and magnetic characteristics.

The characteristics of the CuO NPs rely on the synthesis technique used, and they are crucial for their uses in a variety of fields, the most common of which is biomedical research. The nanoparticles' size, which can be modified during production, is the most crucial component since it enables precise modelling of their optical, catalytic, electrical, and biological capabilities. These characteristics make them ideal for a variety of applications, including the creation of cosmetics, pharmacological substitutes, paints, coatings, and other materials. Antimicrobial drugs are the major use for CuO nanoparticles. They are employed in hospitals because, if the right dose is utilised, they have an antibacterial capacity to kill more than 99.9% of Gram-positive and negative bacteria within two hours of exposure.

## METHOD OF PREPARATION - HYDROTHERMAL METHOD

The most used technique for creating nanomaterials is hydrothermal synthesis. It is essentially a solution-response strategy. The hydrothermal method involves using an aqueous solution as a reaction system in a particular closed reaction vessel to pressurise the reaction system (or the vapour pressure created by itself) and heat it to create a high-temperature, high-pressure reaction environment. The technique dissolves and recrystallizes a chemical that is weakly soluble or insoluble under normal circumstances.

Utilising specialised equipment known as a Hydrothermal Autoclave Reactor is necessary for the hydrothermal approach for synthesising nanoparticles. In order to withstand higher pressures and higher temperatures from within, we need a certain kind of sturdy vessel.

The cylindrical vessels that make up the autoclave reactor have strong steel walls and hermetic seals. Additionally, this enables it to sustain high temperatures and pressures for an extended period of time safely. Furthermore, the autoclave's fabric needs to be solvent-resistant. The 'closing' is perhaps the component of the hydrothermal autoclave reactor that is most important. Moreover, the seals are a further essential component of the autoclave.

Hydrothermal synthesis can produce nanomaterials that are not stable at high temperatures. Nanomaterials with high vapour compression can be produced hydro-thermally with minimal loss of material. The composition of the synthesised nanomaterials can be well controlled by hydrothermal synthesis via chemical solution phase or multiphase reactions.



Parts of Teflon Autoclave used for Hydrothermal Method



# ADOPTIVE CHARACTERISATION METHOD

## FTIR SPECTROMETER

The most popular kind of infrared spectroscopy is known as "FTIR," or "Fourier transform infrared.". All infrared spectroscopies operate under the premise that some IR radiation passes through a sample and is absorbed. It is noted which radiation enters the sample.

These benefits of FTIR come from using a Fourier transform and an interferometer, which serves as the "source" of infrared light and allows for increased speed. The Fourier transform, a mathematical operation, divides waves into their component frequencies, which are then computed in relation to time. Instead of the spectral data we use for spectroscopy, the interferometer's "output" is a graph known as an "interferogram". In order to create the well-known and practical infrared spectroscopy spectrum graph, the Fourier transform is used to transform the interferogram.

In practice, the scans are actually on either side of zero, producing a two-sided interferogram. Mechanical design restrictions may prevent the scan from running to the maximum OPD on both sides of zero for the greatest resolution. The Fourier transformation turns the interferogram into a spectrum. This necessitates that the route difference between the two beams be kept digitally as a sequence of numbers at equal intervals. When a laser beam is sent through an interferometer to measure the path difference, it produces a sinusoidal signal with repeated maxima spaced at intervals equal to the laser's wavelength, which is commonly a 633 nm HeNe laser. By incorporating FTIR into a scanning near-field optical microscope platform, the spatial resolution of FTIR may be increased even more below the micrometre scale. The analogous method, known as nano-FTIR, enables broadband spectroscopy on substances in extremely tiny amounts (single viruses and protein complexes) and with a spatial resolution of 10 to 20 nm.<sup>[6]</sup>



An example of an FTIR spectrometer with an attenuated total reflectance (ATR) attachment

## ZETA POTENTIAL ANALYSER

Zeta potential is a measurement that depends on the charges that the particles in a suspension or emulsion possess. To determine the zeta potential of a certain substance, measurements and computations are performed by a zeta potential analyzer. The product is more stable the greater the zeta potential.

Zeta potential analyzers employ two techniques: applying an electric field and detecting the motion of charged particles, and utilising ultrasonic waves to accelerate charged particles and then measuring their electric charge. The kind of liquids to be analysed, the range of particle sizes that can be measured, the range of zeta potentials that can be measured, and the sample volume that can be accommodated are some factors to consider when comparing zeta potential analyzers.

When the zeta potential of a nanoparticle ranges from -10 and +10 mV, it is assumed to be roughly neutral; however, when it is greater than +30 mV or less than -30 mV, it is considered to be highly cationic or anionic. Because the vast majority of the membranes in life are negatively charged, zeta potential can influence a nanoparticle's ability to pass through surfaces. Cationic particles often exhibit more toxicity due to breakdown of cell walls. This method is illustrated using colloidal gold (highly anionic) and amine-terminated PAMAM dendrimers (strongly cationic), two kinds of nanoparticles frequently employed in biological applications.

Four alternative measuring approaches can be used to assess electrokinetic effects, such as the zeta potential:

- Streaming potential
- Electrophoresis
- Electroosmosis
- Sedimentation potential

The two most popular approaches are electrophoresis and streaming potential, and they are used in equipment like the Litesizer for electrophoresis and the SurPASS for streaming potential, respectively. In general, electrophoretic techniques like electrophoretic light scattering are employed to measure the zeta potential of particles with a diameter of up to 100 nm. Streaming potential is commonly used to determine the zeta potential of larger surfaces (diameter >25 nm).



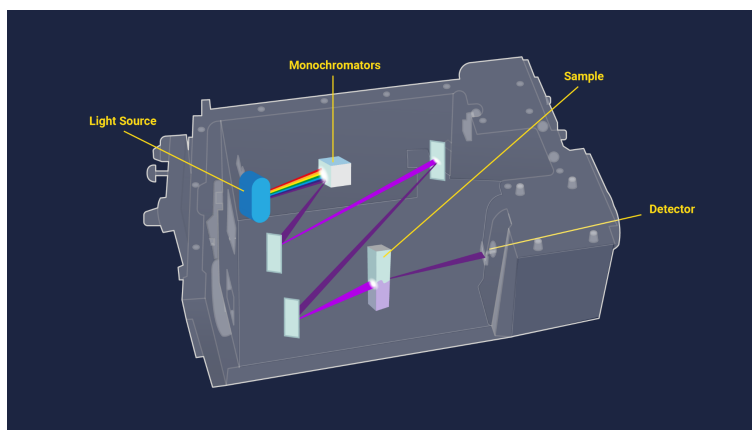
Particle analyzer: Litesizer 500- Anton Paar product used for particle size, refractive index and zeta potential analysing

## UV-VIS SPECTROSCOPY

The terms UV spectroscopy (also known as UV-Vis or UV/Vis) and UV-visible spectrophotometry (also known as UV-Vis or UV/Vis) refer to the absorption and reflectance spectroscopy in the ultraviolet and the full, adjacent visible regions of the electromagnetic spectrum.

The quantitative determination of numerous analytes or samples, including transition metal ions, highly conjugated organic compounds, and biological macromolecules, is frequently carried out in analytical chemistry using UV/Vis spectroscopy. Although solids and gases as well as solutions are frequently studied through spectroscopic analysis. [5]

UV-Vis spectroscopic information can be displayed as plots of absorbance, optical density, or transmittance as a function of wavelength.



Schematic diagram of a cuvette-based UV-Vis spectroscopy system.



Beckman DU640 UV-Vis spectrophotometer:-

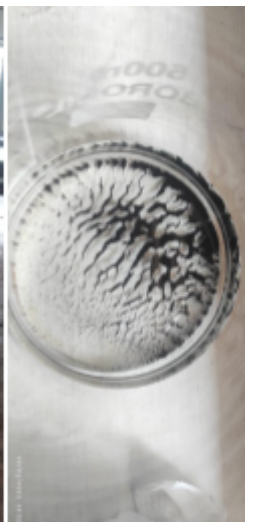
# THE EXPERIMENT

## Chemicals Used

- Copper Malachite [ $\text{CuCo}_3\text{Cu}(\text{OH})_2$ ]
- Ammonium Hydroxide [ $\text{NH}_4\text{OH}$ ]
- Distilled water

## Apparatus Used

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



## Experimental Details and Steps

→ Calculation of mass of Copper Malachite [ $\text{CuCo}_3\text{Cu}(\text{OH})_2$ ]

- Required volume (V) = 50 ml
- Molecular mass of  $\text{CuCo}_3\text{Cu}(\text{OH})_2$  (m) = 221.116 g/mol
- Required molarity of the solution (M) =  $5 \times 10^{-3}$  mol/L
- Mass of  $\text{CuCo}_3\text{Cu}(\text{OH})_2$  (X) =  $\{ m \times M \times V \} / 1000$   
=  $\{ 221.116 \times 5 \times 10^{-3} \times 50 \} / 1000$   
= 0.055279 g

∴ The required mass of  $\text{CuCo}_3\text{Cu}(\text{OH})_2$  is 0.055279 g.

→ STEP 1:

Required amount of  $\text{CuCo}_3\text{Cu}(\text{OH})_2$  is dissolved in distilled water to prepare 50 ml solution of  $5 \times 10^{-3}$  molarity.

→ STEP 2:

It is then mixed with 50 ml distilled water.

→ STEP 3:

The solution is stirred well for 30 minutes.

→ STEP 4:

5 ml of  $\text{NH}_4\text{OH}$  is added to the solution under constant stirring. The solution is vigorously stirred for 1 hour until it turns from aqua blue color to intense blue.

→ STEP 5:

The resultant solution is transferred into a Teflon autoclave and placed in the furnace.

→ STEP 6:

It is then heated to  $180^\circ \text{C}$  for one hour.

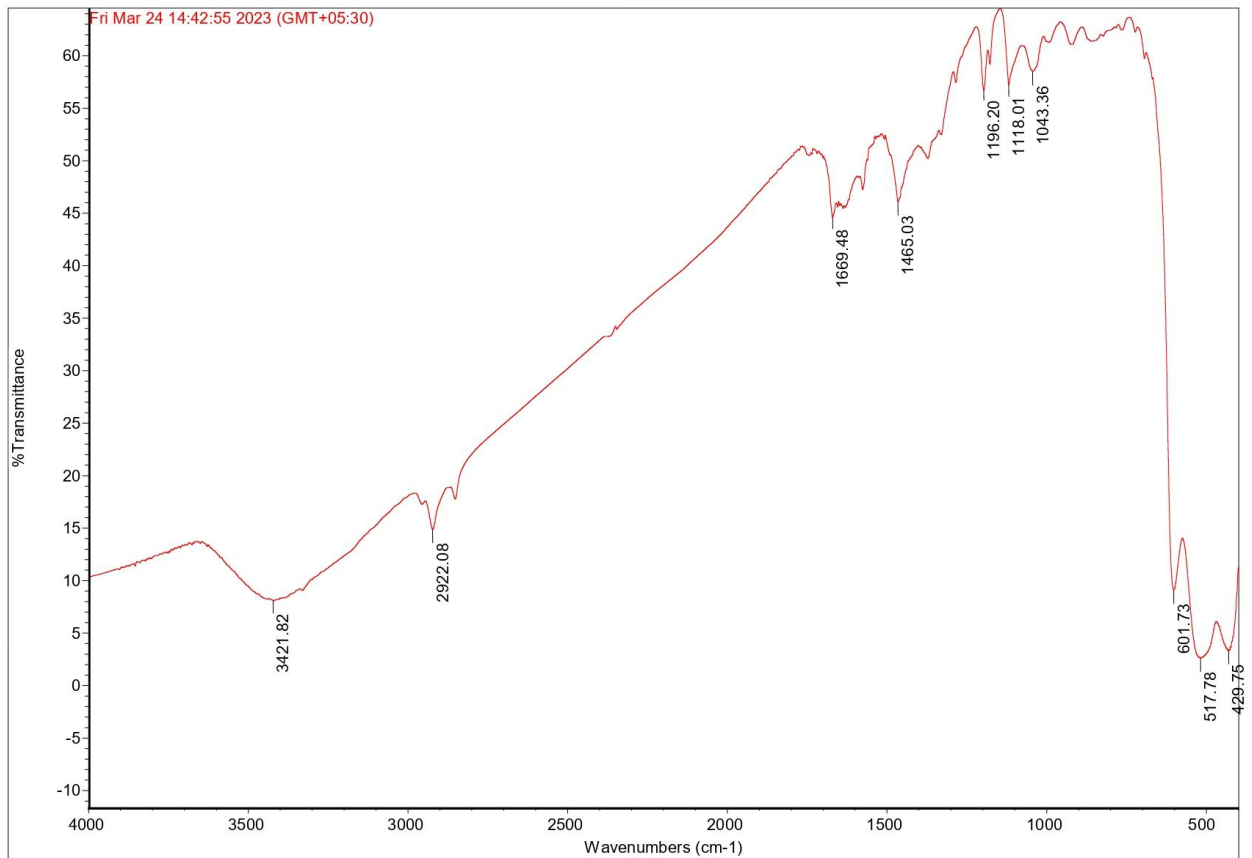
→ STEP 7:

Nanostructures of various sizes are obtained. They are washed several times with distilled water and dried at  $70^\circ \text{C}$  in a hot plate.

Prepared powder were subjected to characterization techniques like FTIR, UV-VIS and Zeta Potential .

## RESULT AND DISCUSSION

### FTIR SPECTRA FOR CuO NANOPARTICLES



The FTIR spectrum of CuO shows peaks at **429.75, 517.78 & 601.73 cm<sup>-1</sup>** revealed the formation of CuO.[\[1\]](#)

## PARTICLE SIZE, REFRACTIVE INDEX AND ZETA POTENTIAL

---

### General

<b>Measurement name</b>	cuo3	<b>User</b>	ADMIN
<b>Method</b>		<b>Time</b>	23-03-2023 15:06:12
<b>Status</b>	Succeeded	<b>Instrument type</b>	Litesizer 500
<b>Measurement type</b>	Particle size		

---

### Settings

<b>Measurement cell</b>	225288 Omega cuvette	<b>Equilibration time</b>	0h 01m 00s
<b>Angle</b>	Automatic	<b>Analysis model</b>	General
<b>Target temperature</b>	25.0 °C	<b>Cumulant model</b>	Advanced

---

### Quality

<b>Quality mode</b>	Automatic	<b>Measurement time</b>	0h 00m 10s
<b>Number of runs</b>	60		

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### Filter

<b>Attenuation mode</b>	Automatic	<b>Attenuation</b>	0
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### Focus

<b>Focus mode</b>	Automatic	<b>Focus position</b>	0.0 mm
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### Material

<b>Name</b>	Unknown material	<b>Refractive index Laser 1</b>	-
<b>Absorption Laser 1</b>	-		

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### Solvent

<b>Name</b>	Water	<b>Refractive index</b>	1.3303
<b>Viscosity</b>	0.0008903 Pa.s		

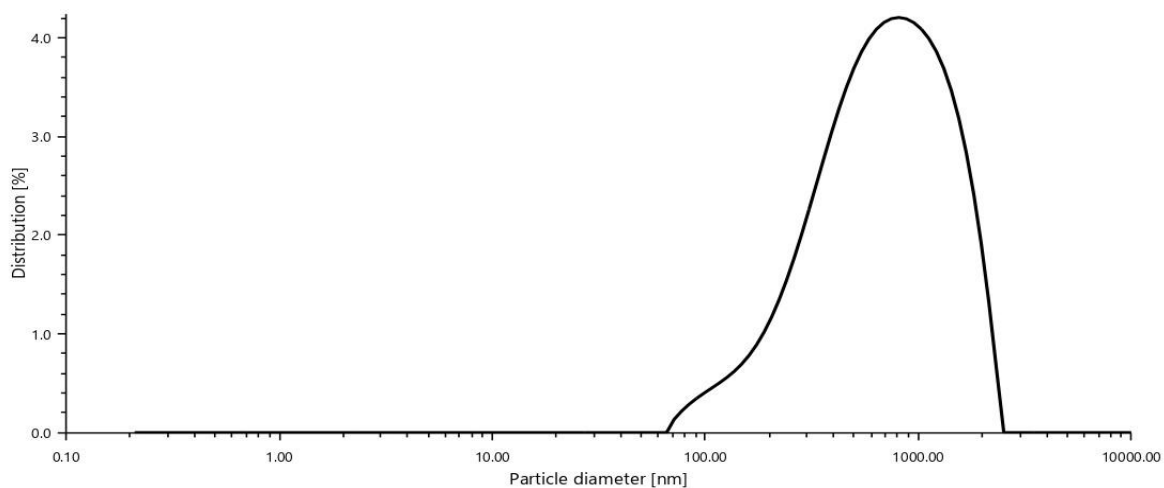
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### Data output

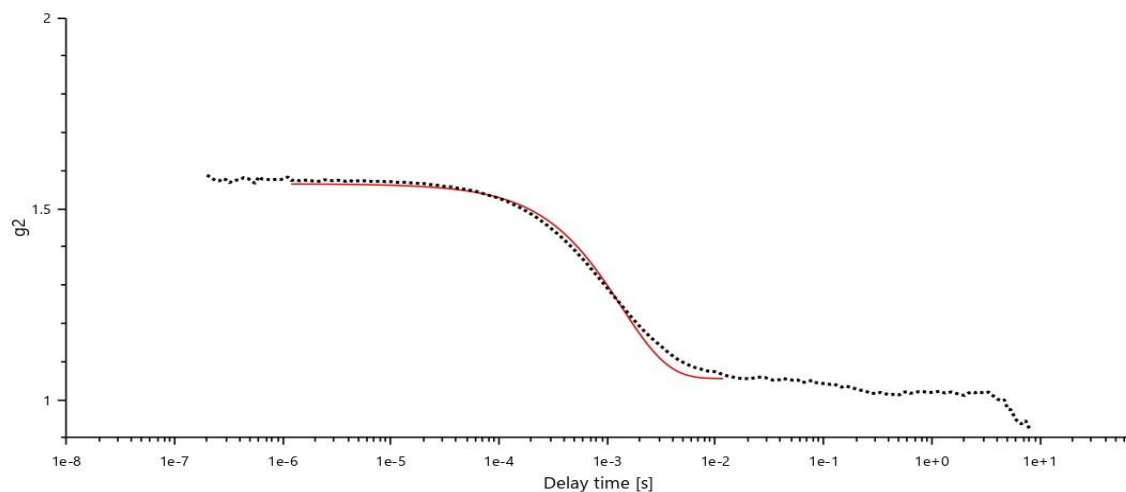
<b>Hydrodynamic diameter</b>	854.8 nm	<b>Polydispersity index</b>	30.3 %
<b>Intercept <math>g1^2</math></b>	0.5099	<b>Mean intensity</b>	224.5 kcounts/s
<b>Filter optical density</b>	2.6049	<b>Baseline</b>	1.055
<b>Focus position</b>	0.0 mm	<b>Angle used</b>	Back scatter
<b>Processed runs</b>	7	<b>Transmittance</b>	82.4 %
<b>Diffusion Coefficient</b>	0.6 $\mu\text{m}^2/\text{s}$		



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**Particle size distribution by intensity**

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**Correlation function**

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**Particle size distribution peaks (intensity)**

	Size [nm]	Area [%]	Standard deviation [nm]
Peak 1	809.7	100.00	640.3
Peak 2	-	-	-
Peak 3	-	-	-

---

**Undersize values**

Size distribution	D <sub>10</sub> [nm]	D <sub>50</sub> [nm]	D <sub>90</sub> [nm]	Undersize span (D <sub>90</sub> -D <sub>10</sub> )/D <sub>50</sub>
Volume	-	-	-	-
Intensity	229.2	665.7	1517.4	1.935
Number	-	-	-	-

---

**Instrument information**

Serial number	82983437	Laser wavelength	658 nm
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**General**

<b>Measurement name</b>	refInd-3	<b>User</b>	ADMIN
<b>Method</b>		<b>Time</b>	23-03-2023 16:01:59
<b>Status</b>	Succeeded	<b>Instrument type</b>	Litesizer 500
<b>Measurement type</b>	Refractive index		

---

**Settings**

<b>Target temperature</b>	25.0 °C	<b>Equilibration time</b>	0h 01m 00s
<b>Measurement time</b>	0h 00m 02s		

---

**Result**

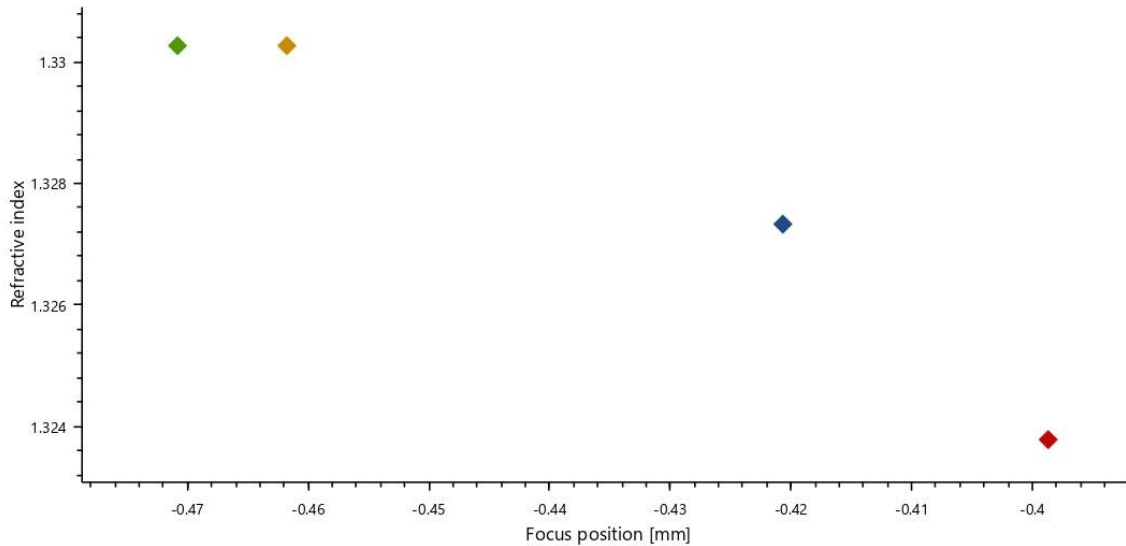
<b>Refractive index</b>	1.327	<b>Fit parameter 1</b>	-1.425
<b>Fit parameter 2</b>	-1.329	<b>Fit parameter 3</b>	1.020

---

**Solvent results**

<b>Solvent</b>	<b>Focus position [mm]</b>	<b>Refractive index</b>
Water	-0.5	1.330
Water	-0.5	1.330
Methanol	-0.4	1.324
Unknown	-0.4	1.327

---

**Focus position over refractive index****Instrument information**

<b>Serial number</b>	82983437	<b>Laser wavelength</b>	658 nm
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**General**

<b>Measurement name</b>	cuo zeta - 2	<b>User</b>	ADMIN
<b>Method</b>		<b>Time</b>	23-03-2023 15:14:36
<b>Status</b>	Succeeded	<b>Instrument type</b>	Litesizer 500
<b>Measurement type</b>	Zeta potential		

---

**Settings**

<b>Measurement cell</b>	225288 Omega cuvette	<b>Equilibration time</b>	0h 01m 00s
<b>Approximation</b>	Smoluchowski	<b>Target temperature</b>	25.0 °C
<b>Adjustment mode</b>	Automatic	<b>Voltage</b>	200.0 V
<b>Henry factor</b>	1.5		

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**Quality**

<b>Run mode</b>	Automatic	<b>Number of runs</b>	1000
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**Solvent**

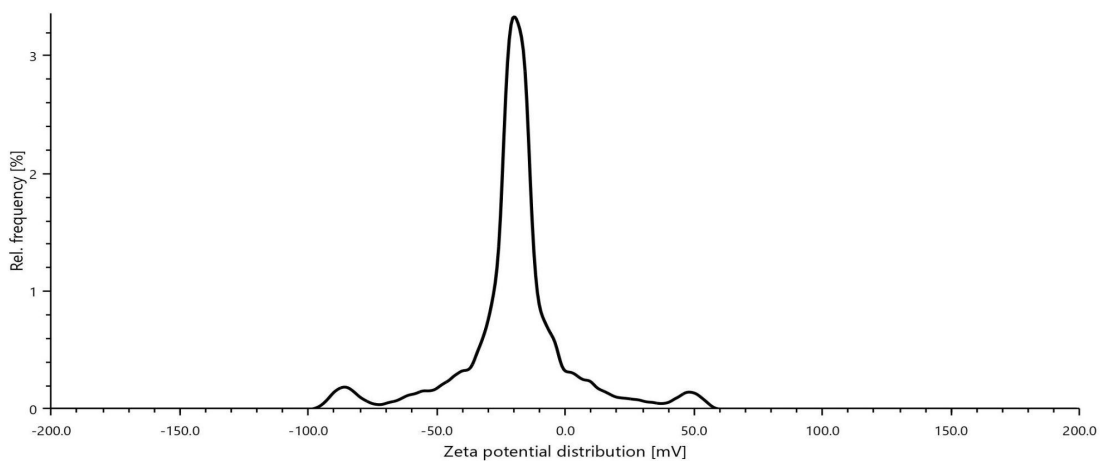
<b>Name</b>	Water	<b>Refractive index</b>	1.3303
<b>Viscosity</b>	0.0008903 Pa.s	<b>Relative permittivity</b>	78.37

---

**Data output**

<b>Mean zeta potential</b>	-19.9 mV	<b>Electrophoretic mobility</b>	-1.5503 $\mu\text{m}^2\text{cm/Vs}$
<b>Distribution peak value</b>	-19.8 mV	<b>Filter optical density</b>	1.6070
<b>Processed runs</b>	100	<b>Conductivity</b>	0.005 mS/cm
<b>Adjusted voltage</b>	200.0 V	<b>+/- Standard deviation</b>	0.7 mV
<b>Mean intensity</b>	785.7 kcounts/s	<b>Transmittance</b>	83.5 %

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**Zeta potential distribution**

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**Instrument information**

<b>Serial number</b>	82983437	<b>Laser wavelength</b>	658 nm
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The Mean Zeta potential of CuO nanoparticles dispersed in water was found to be **-19.4 mV**.[\[2\]](#)

## UV-VIS SPECTRA

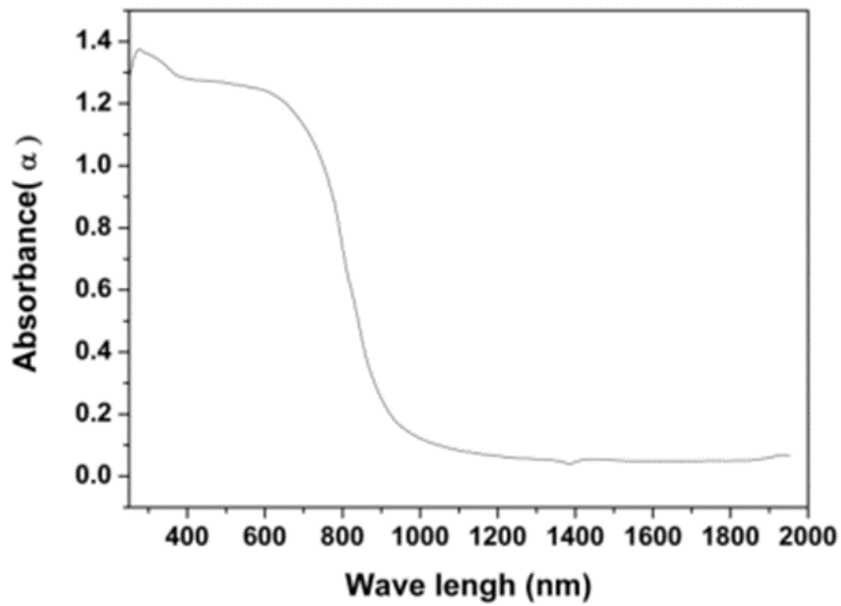


Figure 1: UV-VIS absorbance spectrum

Figure 1 and 2 show the absorbance and reflectance curves obtained from UV-VIS spectra. More than 60% absorbance was observed for wavelength from 250 to 800 nm.[\[3\]](#)

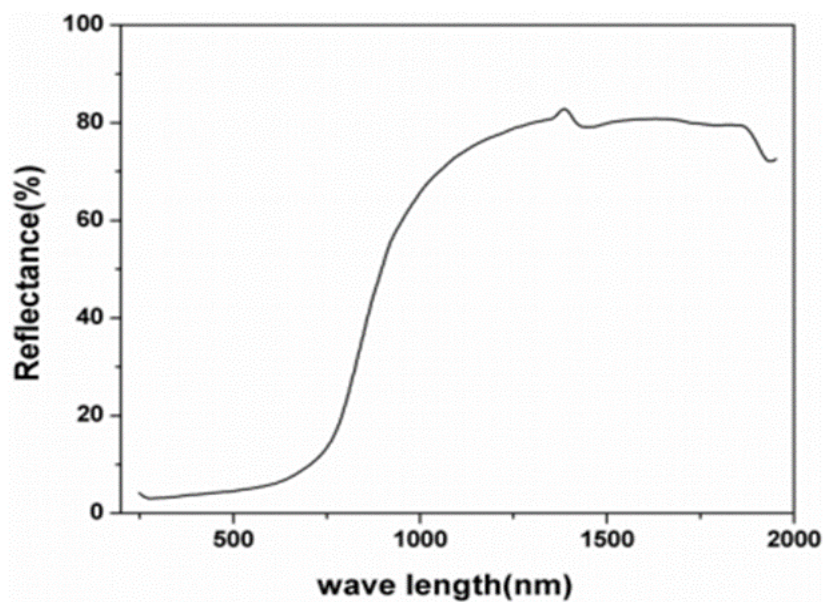
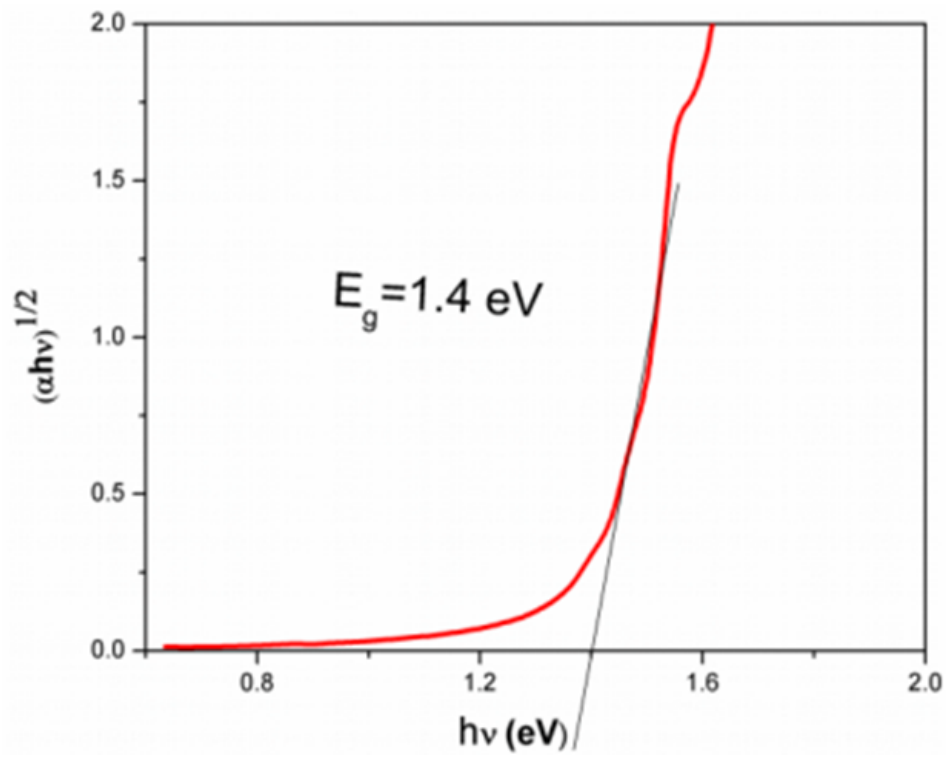


Figure2: UV-VIS reflectance spectrum.



The calculated band gap value was **1.4eV**.[\[4\]](#)

## CONCLUSION

- CuO nanoparticles were synthesised using the Hydrothermal Method.
- Prepared sample of CuO is confirmed by FTIR.
- The size varied considerably due to some impure contents in the sample.
- Zeta potential, UV-VIS spectrum, and Optical band gap proved the CuO properties for optical characteristics.

# REFERENCES

- [1]- <https://www.researchgate.net>
- [2]-[https://www.researchgate.net/publication/277640820\\_Green\\_Synthesis\\_of\\_Cupric\\_Oxide\\_Nanoparticles\\_Using\\_Water\\_Extract\\_of\\_Murrya\\_koenigi\\_and\\_its\\_Photocatalytic\\_Activity](https://www.researchgate.net/publication/277640820_Green_Synthesis_of_Cupric_Oxide_Nanoparticles_Using_Water_Extract_of_Murrya_koenigi_and_its_Photocatalytic_Activity)
- [3]-<https://www.sciencedirect.com/science/article/abs/pii/S1386142514004387?via%3Dihub>
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- <https://www.sciencedirect.com/topics/materials-science/optical-property-of-nanomaterials>
- <https://www.ncbi.nlm.nih.gov>
  
- Flower like CuO synthesization of CuO flower nanostructures (college of science China)
- Synthesis and Characterization of CuO flower nanostructures (Department of physics, Brazil university)
- Hydrothermal Synthesis of CuO nanostructures and their Characterisation (Department of Physics, Annamalai University)