



Synthesis of CuO/NaCuSO₄ nanocomposite using an aqueous extract of *Tribulus Terrestris* and their structural, optical, morphology and dielectric studies

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Abstract

The development of an environment-friendly process for synthesizing nanoparticles in the field of bionanotechnology is growing day-by-day. Increasing drug resistance in microbes has compelled researchers to synthesize biologically active nanoparticles. In this study, we used *Tribulus terrestris* plant extract to synthesize CuO/NaCuSO₄ nanocomposite. The synthesized samples were characterized through functional groups analysis of FTIR and morphological analysis of SEM. The X-ray diffraction (XRD) and ultra-visible light absorbance analysis (UV–vis) were used to find the nanocrystalline nature and bandgap energy of the biosynthesized copper oxide (CuO) nanoparticles, respectively. In the metal oxide region of Fourier transform infrared (FTIR) spectroscopy the copper oxide nanoparticles were confirmed at 523 cm^{−1} which showed the nature of plant extracts to control over the reduction and stabilizing of the nanoparticles. The Rietveld refinement analysis yielded unit cell compositions of 53.39% for CuO and 46.61% for NaCuSO₄, respectively. The *monoclinic structure* of CuO was confirmed, and the crystalline size was calculated as 37 nm by XRD analysis. From UV–vis spectroscopy, the absorption peak was observed at 277 and 337 nm, revealing the presence of CuO nanoparticles. In fact, the cluster-like morphology patterns are captured by scanning electron microscopy (SEM) and particle sizes were observed around 102 nm. Finally, the dielectric properties of the synthesized copper oxide nanoparticles have been examined and reported in detail.

Keywords Electrical conductivity · Insulator · Dielectric studies · UV-vis spectroscopy · Phase transition · NaCuSO₄ nanoparticles

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Introduction

Nanotechnology is an area of science and technology concerned with the creation of substances smaller than 100 nm in size (Krithiga et al. 2015; Arya et al. 2018). Nanomaterials are differentiated into three types, these are classified as natural nanomaterials, incidental nanomaterials, and engineered nanomaterials (Barhoum et al. 2022). Natural nanomaterials exist in nature for example smoke, volcanic dust, and bacteria (Griffin et al. 2017). Incidental nanomaterials are unintended by-products of human activity, for example, diesel exhaust, coal combustion, and welding fumes (Nasrollahzadeh et al. 2019). In engineered nanomaterials, they are intentionally created with structures like nanotubes and fullerenes. The engineered nanomaterials are synthesized in two ways. The first way is a top-down approach, and the next way is bottom-up approach (Iqbal et al. 2012). The top-down approach is suitable for bulk industrial production, but nanomaterials synthesized by this way may possess crystal

imperfections and surface imperfections. Mechanical grinding or ball milling is the most preferred method for the top-down approach (Arole and Munde 2014). The exact opposite method is the bottom-up approach, this is sort of starting from the atomic scale at or nearer to the atomic size scale (Nasrollahzadeh et al. 2020). The bottom-up approach provides us the nanoparticles with identical size or mono-sized, identical shape, identical composition, and individually dispersed or mono-dispersed (Sahooli et al. 2012; Chokkareddy and Redhi et al. (2018). Metal nanoparticles have gained attention in recent decades due to their increased area by weight or volume, and various qualities such as electrical (Kumar et al. 2018), , magnetic (Lakshmanan et al. 2021), optical (Azam et al. 2012), biological, and chemical properties Javed et al. 2017) have attracted researchers to this topic. In general, the emergence of infectious diseases poses a threat to public health around the world, particularly with the rise of antibiotic- strain resistant (Anu et al. 2020). Gram-negative and gram-positive bacterial strains are considered serious health risks, so antibiotics were used to treat the ensuing infections (Pfeiffer et al. 2014).

In Mesopotamia in the ninth century, the coloring of glass and ceramics was one of the first applications of copper nanoparticles (Heiligtag and Niederberger 2013). This was accomplished by making an enamel coating out of salts containing copper and silver and applying it to clay pottery. Metal ions moved outside the glaze and were reduced to metals when the ceramic was burned at a high temperature under reducing circumstances. The first layer of the final ceramic allowed light to pass through and reflect from the surface. The second layer of nanoparticles reflects the light that enters the first layer and interferes with the light that is reflected on the first layer, producing an interference effect that is both constructive and destructive and results in a luminous effect (Deshmukh 2019). In the same way, stained glass was also made of silver and gold nanoparticles. Another historical example is the Lycurgus cup. It is a roman glass gauge cup made of dichroic glass with silver and gold nanoparticles (Dekker et al. 2020). Copper oxide is an enticing *p*-type metal oxide semiconductor with distinct optical, electrical, and catalytic characteristics (Patel et al. 2013). According to the consensus, copper oxide nanoparticles have remarkably unique physicochemical features. Catalysis, nanotherms, oxidants, electrical, optical, and biological applications all made use of it. Since copper and its alloys can destroy 99.9% of bacteria in just two hours, the United States Environmental Protection Agency (USEPA) has also permitted their use as solid surface materials on tangible surfaces (Kumar et al. 2023a).

Copper nanoparticles with high catalytic activity can be applied to biosensors and electrochemical sensors. The redox reactions used in these sensors are generally irreversible and require high over potentials (*more energy*) to

function. Indeed, nanoparticles have the ability to reverse redox reactions and reduce over potentials when applied to sensors. Another application of copper nanoparticles is the glucose sensor (Ibupoto et al. 2013; Kumar et al. 2023b). When we use the nanoparticle frames, the sensor does not need any enzymes, and therefore, it is not affected by the denaturation and degradation of the enzymes. The nanoparticles allow the sensor to be more stable and more resistant to toxic chemicals at higher temperatures and variable pH. Furthermore, using nanoparticles, it is possible to detect native amino acids. A carbon electrode screen printed with copper nanoparticles serves as a stable and efficient detection system to detect all 20 amino acids. The title plant information was gathered from its botanical history Zygophyllaceae family, Tribulus genus, Species: *Terrestris* Linn. It is generally known as *Goshur* and called *Puncture vine* (Saurabh et al. 2012). It has more than 20 species in its Zygophyllaceae family around the world. Traditionally *Tribulus terrestris* was used to prepare tonics in folk medicines, to treat urinary disinfectant, antihypertensive, palliative, lithotriptic, and diuretic (Parimala et al. 2021).

The phytochemical screening of *Tribulus terrestris* found that tannins, glycosides, alkaloids, saponins, and flavonoids were all present in the plant. The literature reports that the plant *Tribulus terrestris* has variable saponin composition in various geographical regions. *Tribulus terrestris* has diuretic qualities for an abundance of nitrates and essential oil found in its fruits and seeds. Potassium salts which are present in large quantities, can also be attributed to the diuretic effect (Parimala et al. 2021). The similar work observed from the literatures, the synthesis of CuO nanoparticles by using the plant extract of *moringa oleifera* and the green synthesis of copper oxide nanoparticles by *spinacia oleracea* leaf extract. Some of the previously reported copper oxide nanoparticles can be listed in Table 1.

There are several techniques used to synthesize copper oxide nanoparticles such as chemical precipitation, thermal decomposition, precipitation pyrolysis, sol–gel method, sonochemical method and microwave irradiation (Thakur et al. 2022). Even though these types of methods to synthesize nanoparticles offer a high quantity of yields, the use of this method is highly toxic and produces unwanted by-products. Recent research has shown that natural biomasses and biological substances can be used for synthesizing nanoparticles. The biosynthesis of nanoparticles is a process that can be used to create safer, more affordable, and healthier alternatives for the environment and human health (Thakur et al. 2021). Electronic components and wireless networks have been made by using transition metal oxide due to its higher dielectric constant, and low dielectric loss. Among these metal oxides, copper nanocrystals have attracted due to their electrical and surface-to-volume ratio (*s/v*) driven features. In this work, we report the synthesis of copper

Table 1 Previous literature of biosynthesized metal oxide nanoparticles

S. No	Name of the plant	Plant material	Metal oxide nanoparticles	References
1	<i>Moringa Oleifera</i>	Leaves	CuO	Ikhioya et al. (2023)
2	<i>Couroupita guianensis</i>	Leaves, stems, petals, and bark	CuO	Logambal et al. (2022)
3	<i>Bougainvillea</i>	Leaves	CuO	Al-Fa'ouri et al. (2023)
4	<i>Tamarix gallica</i>	Leaves	CuO	Nasrollahzadeh et al. (2015a)
5	<i>Euphorbia chamaesyce</i>	Leaves	CuO	Maham et al. (2017)
6	<i>Euphorbia polygonifolia</i>	Leaves	Fe ₃ O ₄ @CuO	Pakzad et al. (2020)
7	<i>Anthemis xylopoda</i>	Flowers	Cu	Nasrollahzadeh et al. (2015b)
8	<i>Azadirachta indica</i>	Leaves	Fe ₂ O ₃	Kumar et al. (2023c)
9	<i>Acroptilon repens</i>	Flower	SnO ₂ —ZnO/bentonite nanocomposite	Golmohammadi et al. (2023a)
10	<i>Melia Azedarach</i> fruit	Fruit	Ag NPs	Golmohammadi et al. (2023b)

oxide nanoparticles by using the plant extract of *Tribulus terrestris*. The extract acts as a reducing agent as well as a capping agent to control the size and increase the stability of the nanoparticles. The synthesized nanoparticles were characterized by different analytical techniques. Further, we focused on exploring the dielectric properties of copper oxide nanoparticles at room temperature, we examined the frequency-dependent dielectric constant.

Experimental procedure

Materials

The healthy and fresh plant of *Tribulus terrestris* was harvested from the Tiruvannamalai district, Tamil Nadu, India. To properly rinse off all the dust and other annoying visible particles, it was split into little pieces and thoroughly cleaned with normal water and then deionized water. The washed plant was shadow dried to avoid direct drying from sun rays because of the phytochemicals, which may destroy the direct drying. The shade-dried plants were crushed into a fine powder and stored in a moisture-free container for further aqueous extract preparation. Copper sulfate anhydrous [CuSO₄] and sodium hydroxide [NaOH] were purchased with an analytical grade which was used directly without any further purification. All the experimental work was done with sterile distilled water.

Tribulus terrestris plant extract preparation

The 10 g of crushed fine powder of *Tribulus terrestris* was taken in a 250 ml borosilicate beaker, and 100 ml of double distilled water was added into the beaker. It was boiled for 15 min at 80 °C. The color of the leaf solution turns brown

color. Then the prepared aqueous extract was filtered using Whatman filter paper no.1 and stored in a refrigerator under 5 °C for further synthesis.

Green synthesis of copper oxide nanoparticles

The copper oxide nanoparticles were synthesized by the co-precipitation method using *Tribulus terrestris* aqueous extract. In a 250 ml borosilicate beaker, 0.1 M of copper sulfate anhydrous [CuSO₄S] was dissolved in 80 ml of double distilled water as shown in Fig. 1a. The mixtures of salt were continuously stirred, and 20 ml of prepared *Tribulus terrestris* aqueous extract as shown in Fig. 1b was added into the stirring solution, and the temperature was maintained at 60 °C. After stirring for 1 h, 0.1 M of NaOH solution was added to the mixture solution. Then it was kept in a continuous stirring process for up to 3 h, and the pH value was maintained at ~ 12. After a 3 h continuous stirring process, the color of the solution was changed from blue to darkish green as shown in Fig. 1c which optically confirms the formation of CuO nanoparticles. The prepared solution was kept until aging to overnight, and this was centrifuged and washed several times using ethanol and acetone. The precipitate was dried and calcinated at 500 °C for 2 h. Finally, greenish black fine powder was collected and stored in a moisture-free sample holder to carry out further research work.

Analytical methods for the green synthesized CuO nanoparticles

The biologically synthesized copper oxide nanoparticles were characterized by different analytical techniques namely UV–vis, FTIR, XRD, DLS and SEM analysis.

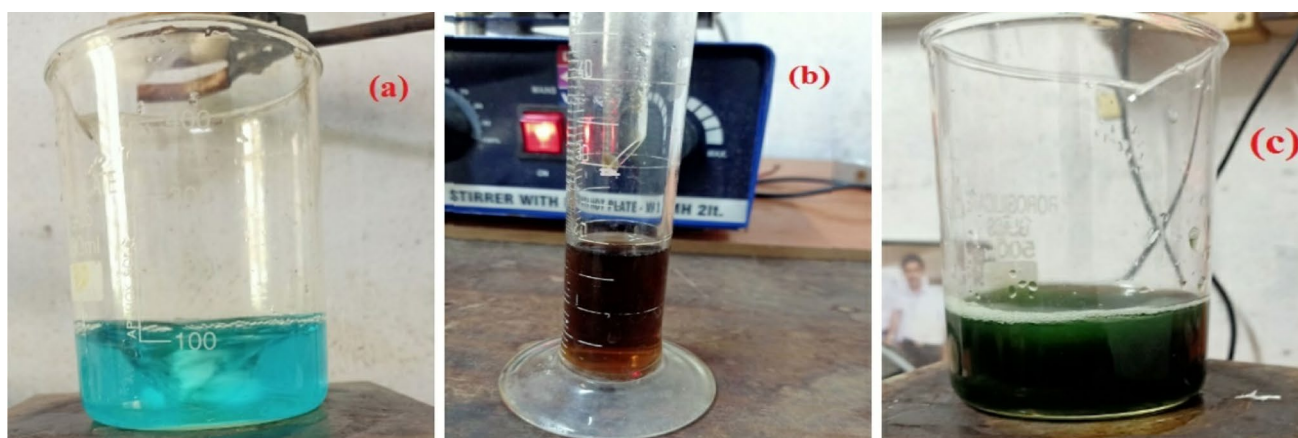


Fig. 1 **a** Solution of copper sulfate; **b** Extract of *Tribulus terrestris*; **c** CuO nanoparticles solution

UV–vis spectroscopy

Initially, the copper oxide nanoparticles were analyzed by Ultraviolet and visible (UV–vis) absorption spectroscopy. Perkin Elmer–Lambda 35 model spectrophotometer was used to analyze the sample at 200 nm to 800 nm wavelength range. It is the attenuation of a beam of light after passing it through a sample or reflection from a sample surface. This spectroscopic technique is used to find the optical properties and energy gap of the synthesized nanoparticles.

FTIR analysis

The functional elements are identified by the Perkin Elmer—spectrum two model of the Fourier transform infrared spectrometer. It is an analytical technique used to identify whether the synthesized sample materials are organic, polymeric, or in some other cases inorganic materials or not. The functional materials can be analyzed in the frequency range from 4000 to 400 cm^{-1} . It measures the absorption of infrared radiation by the sample material at different ranges. Generally, the metal oxides are observed in the fingerprint region at lower frequencies. The infrared absorption bands are also used to identify molecular band vibration and structure of the synthesized sample material.

Powder X-ray diffraction technique

The powder X-ray diffraction analysis was used to study the structural information of the synthesized sample. This analysis was done by using Bruker—D8 advanced powder X-ray diffractometer. 2.2 KW Cu-anode ceramic tube was used as an X-ray source and the diffracted rays were detected at the angle range of 10° to 90° by Lynx eye detector (silicon strip detector technology) and scintillation detector (for low angle XRD analysis). By importing the experimental pattern

of PXRD data into the Rietveld refinement software, the detailed structural information of the synthesized sample material can be studied.

DLS analysis

By the dynamic light scattering analysis (DLS) technique, the size distribution of particles in synthesized copper oxide nanoparticles was investigated and the size of a particle was studied using micromeritics—nano-plus particle size analyzer. It can measure the samples in the dilution method. The samples are suspended in liquids from the range of 12.3 μm to 0.1 nm with a concentration of 40% to 0.00001% and the sensitivity of a molecular weight range of as low as 250 Da.

SEM analysis

The CAREL ZEISS-EVO 18 scanning electron microscope was used to study the morphological information of the synthesized—sample. The surface morphology of the nanoparticles can be identified by the fast—moving electrons with wave-like characters, which allowed for scanning on a two-dimensional surface. The true environmental SEM allows the samples to be examined in their natural state under the condition of very high-water vapor pressure of up to 3000 Pa. A scanning electron microscope (SEM) scans using a focused electron beam over a surface to create an image. The electrons in the beam interact with the sample producing various signals that can be used to obtain information about the surface topography and composition.

Broadband dielectric spectroscopy analysis

The broadband dielectric spectroscopy technique was used to examine the dielectric characteristics of the synthesized materials such as dielectric constant and dielectric loss.

This was done by using an instrument NOVO-CONTROL Technologies—80. Dielectric spectroscopy is also known as impedance spectroscopy which measures the dielectric behavior of a medium as a function of frequency. Initially, alternating electric field frequencies are applied to the sample material by placing it between two electrodes and the output is monitored based on the applied frequencies. It is based on the interaction of an external field with the electric dipole moment of the sample often expressed by the permittivity. The interaction of electromagnetic waves with matter in the frequency range of 10^{-6} to 10^{12} Hz is the domain of broadband dielectric spectroscopy.

Results and discussion

UV–vis spectroscopy

UV–vis spectroscopy is a kind of absorption spectroscopy. A sample is irradiated with electromagnetic waves of different wavelengths in the ultraviolet and as well as visible regions. The sample may absorb a certain amount of UV or visible light depending on the sample material characteristics. The remaining light will be transmitted, an appropriate detector records the transmitted light or the residual light. The sample's distinctive UV–vis spectrum is subsequently generated by the detector. Prior to this, we confirmed the formation of copper oxide nanoparticles by optical color transformation of the colloidal solution. During the synthesis process sky, the blue-colored colloidal solution turned greenish black, which confirms the formation of copper oxide nanoparticles. Generally, the copper oxide nanoparticles display absorption

in the range of 250 nm–300 nm. The biosynthesized CuO nanoparticles are analyzed by UV–vis spectrophotometer, and the absorption peaks are observed at 277 nm and 337 nm as shown in Fig. 2a. The energy bandgap of the biosynthesized CuO nanoparticles was calculated by the following equation.

$$E_g = h\nu_g = \frac{hc}{\lambda_g} \quad (1)$$

where h , C , and λ_g are the plank's constant ($h = 6.623 \times 10^{-34}$ J/s), the velocity of light ($C = 2.99 \times 10^8$ m/s), and the wavelength observed in maximum absorption peak (277 nm and 337 nm), respectively. Using the above equation, the energy bandgap of CuO nanoparticles has been determined, and the calculated value is 3.6 eV. The bandgap energy was calculated by the *Tauc plot* method as 3.22 eV as shown in Fig. 2b. The calculated value is exactly equal to the value of pure copper oxide nanoparticles.

FTIR analysis

The Fourier transform infrared spectroscopy analysis was used to identify the phytochemicals of *Tribulus terrestris*, which is responsible for reducing, forming, and stabilizing the copper oxide nanoparticles. It measures the absorption of infrared radiation by the sample. Infrared radiation absorption refers to identifying the molecular band vibration and the structure. A sample is exposed to infrared light from the FTIR instrument in the range of 4000 cm^{-1} to 400 cm^{-1} . Some part of the radiation is absorbed, and some is sent through. The absorbed

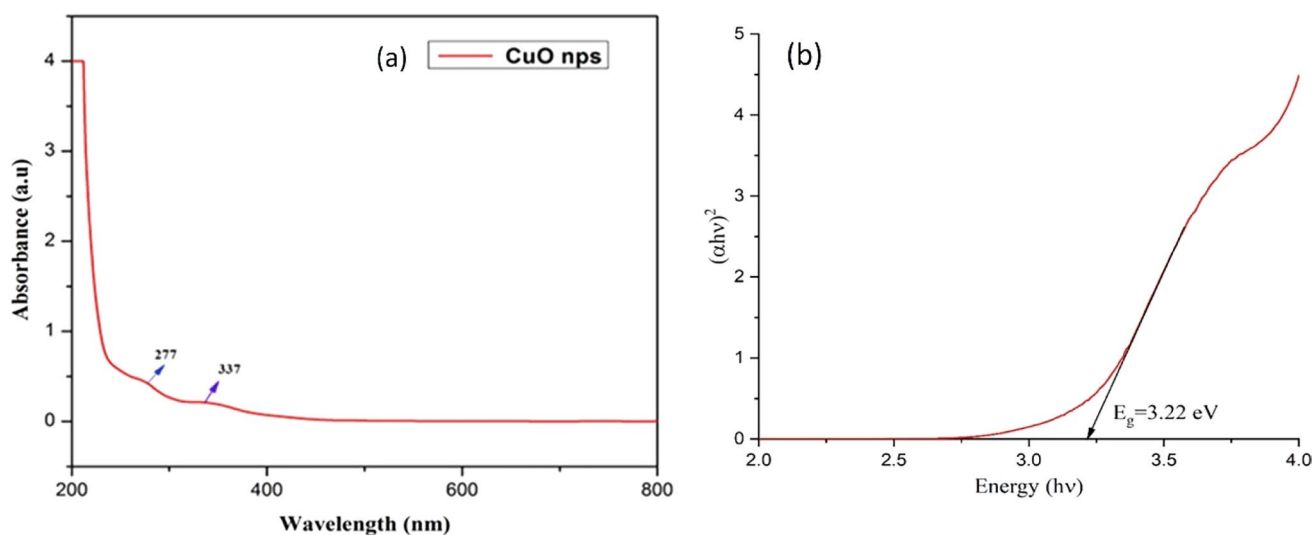


Fig. 2 **a** UV–vis absorbance spectrum; **b** The bandgap energy was calculated by the *Tauc plot* method of CuO nanocomposite using an aqueous extract of *Tribulus Terrestris*

IR ray was converted into rotational and/or vibrational energy. At the detector, the resulting signal appears as a spectrum typically ranging from 4000 to 400 cm^{-1} , which represents the sample's molecular fingerprint. FTIR is a fantastic tool for identifying specific chemical components. Because each molecule structure will generate a distinct spectral fingerprint region which is used to easily identify the organic, inorganic, polymeric, or some other materials and their chemical properties. The observed peaks of possible biomolecules are analyzed and shown in Fig. 3. The intense peaks of biosynthesized CuO nanoparticles were located at 3747 cm^{-1} , 3422 cm^{-1} , 2925 cm^{-1} , 2852 cm^{-1} , 2023 cm^{-1} , 1627 cm^{-1} , 1381 cm^{-1} , 1100 cm^{-1} , 783 cm^{-1} , 595 cm^{-1} and 523 cm^{-1} which are listed in Table 2. The peak at 3747 cm^{-1} is assigned to the O–H stretching vibration of hydroxyl groups, and the broad band observed at 3422 cm^{-1} represents the O–H stretching of alcohol and phenolic compounds. The peak at 2925 cm^{-1}

is attributed to the C=C stretching of alkenes groups, and the absorption peak at 2852 cm^{-1} corresponds to the C–H stretching of aldehyde (*carboxylic acid*). The band at 2023 cm^{-1} represents the C–H bending vibration of aromatics and 1627 cm^{-1} shows the C=O stretching of amides. The peak at 1381 cm^{-1} and 1100 cm^{-1} are assigned to the C–H stretching of alkanes and the C–N stretching of aliphatic amines. In the metal oxide region, the narrow bands are observed at 783 cm^{-1} , 595 cm^{-1} , and 523 cm^{-1} . The peak at 783 cm^{-1} denotes C–H bending of aromatics. The narrow bands 595 cm^{-1} and 523 cm^{-1} are assigned to the mode of CuO lattice stretching, which confirms the formation of copper oxide nanoparticles. The screening of functional groups analysis was done, and it shows that the biomolecules act a major role in the effective protection and formation of copper oxide nanoparticles.

XRD analysis

A method that is used for examining the atomic or crystalline structure of a powder sample is called X-ray diffraction. By the way, it was employed to gather the crystalline phases of the CuO nanomaterial and so expose the information of chemical compositions to examine the nature of the crystal structure. Comparing the collected data to that in the reference database allows for phase identification. Evaluation of minerals, polymers, corrosion byproducts, and unidentified materials can be done by X-ray diffraction. To conduct this experiment, an X-ray beam is pointed at a sample, and the intensity of the scattered radiation is measured. Bragg law is used for finding the 'd' spacing parameters, which is given by Dinneber and Billinge et al. (2019).

$$n\lambda = 2d \sin \theta \quad (2)$$

where n, λ , d, θ are denoted as an integer ($n = 1, 2, 3$, etc.), the wavelength of the X-rays in angstroms, interatomic spacing in angstroms, and the diffracted angle in

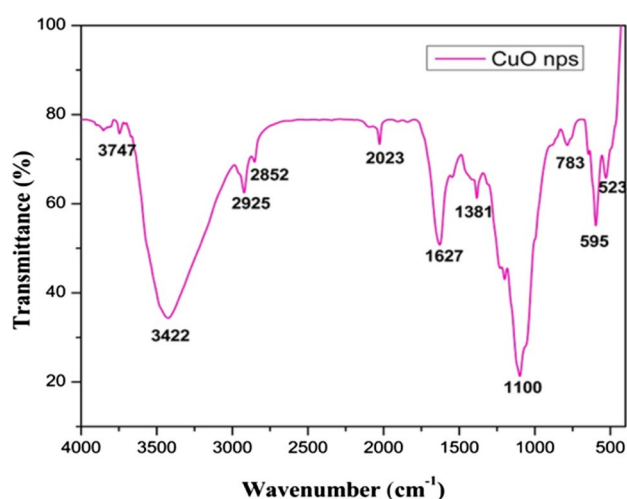


Fig. 3 FTIR analysis of CuO nanocomposite using an aqueous extract of *Tribulus Terrestris*

Table 2 FTIR analysis different functional groups

S. No.	Observed peak position (cm^{-1})	Mode of vibration	Functional group	References
1	3747	O–H stretching	Hydroxyl groups	(Imran et al. 2019)
2	3422	O–H stretching	Alcohol, phenol groups	(Mani et al. 2019)
3	2925	C=C stretching	Alkenes	(Sun et al. 2014)
4	2852	C–H stretching	Aldehyde (carboxylic acid)	(Prasannaraj and Venkatachalam et al. 2017)
5	2023	C–H bending	Aromatic compounds	(Alahmad et al. 2022)
6	1627	C=O stretching	Amides	(Ismail et al. 2014)
7	1381	CH	Alkanes	(Mani et al. 2020)
8	1100	C–N stretching	Aliphatic amine	(Ravichandran et al. 2016)
9	783	Sp_2 CH bending	Aromatics	(Bock and Gierlinger et al. 2019)
10	595 & 523	$\delta(\text{O–C=O})$ stretching vibrations	Cu–O bonds (CuO NPs)	(Phiwdang et al. 2013)

degree, respectively. XRD result indicates the presence of Cu and O in the biosynthesized sample, and it is responsible for fine peaks with intense. The crystallite's dimensional distribution (D) was calculated by Debye–Scherrer equation (Eq. 5). *Hugo M. Rietveld* proposed the Rietveld refinement technique for refining the XRD pattern. Fullprof software was used to find the crystal parameters. It gives the complete information about the sample like crystalline size, structure, cell parameters, chemical compositions etc, (Falsafi et al. 2020). After performing Rietveld refinement, two types of crystalline structures have been confirmed in the synthesized sample and it was found as CuO and NaCuSO₄ nanomaterials. From the CuO nanoparticles, the diffraction peaks observed at 32.53°, 35.47°, 38.75°, 48.78°, 53.51°, 58.34°, 61.60°, 66.30°, 68.15°, 72.46°, 75.31° are attributed to the miller indices of (110), (002), (111), (202), (020), (202), (113), (311), (220), (311) and (222), respectively, and the lattice parameters of CuO nanoparticles are tabulated in Table 3. This confirms that the CuO nanoparticles can be synthesized from the stabilization of copper sulfate with *Tribulus terrestris* extract. The observed cell parameters of the biologically synthesized CuO nanoparticles are $a = 4.68318 \text{ \AA}$, $b = 3.42184 \text{ \AA}$, and $c = 5.12653 \text{ \AA}$ and the coaxial angle values are found with $\alpha = \gamma = 90^\circ$ and $\beta = 99.49068^\circ$. These values indicate that the CuO nanoparticles have the nature of a monoclinic crystal structure. From the Rietveld software, it was named a tenorite crystal structure. The peaks in the XRD pattern match with the data card similarly, a new structure of NaCuSO₄ was observed in the biosynthesized sample. The creation of a new structure takes place due to the interaction of copper sulfate with sodium hydroxide during the synthesis process. Its unit cell parameters are calculated as $a = 8.82040 \text{ \AA}$, $b = 6.19115 \text{ \AA}$, and $c = 7.50736 \text{ \AA}$ (as shown in Table 4). According to the literature data, this type of new crystal structure was rarely reported. It has strong intense peaks and the NaCuSO₄ monoclinic (*natrochalcite*) crystal structure attributed to the online data entry (Entry No: 96-900-4182). The Rietveld refined XRD pattern of CuO and NaCuSO₄ (TTC) graph was plotted as shown in Fig. 4. From the Rietveld refinement analysis, the unit cell compositions of CuO and NaCuSO₄ were calculated as 53.39% and 46.61%, respectively. The cartesian coordinates of the x, y, and z positions of CuO and NaCuSO₄ are tabulated in Table 4. The two-dimensional (2D) and three-dimensional (3D) structure views of CuO

Table 4 Cartesian coordinates position occupied by CuO and NaCuSO₄ atomic units with monoclinic structure in the unit cell box ranges, $x = -1, 1$; $y = -1, 1$ and $z = 0, 1$

Atom	Cartesian coordinates position		
	X	Y	z
<i>CuO</i>			
Cu ₁	0.25000	0.24680	0.00000
O ₂	− 0.01150	0.41710	0.25370
<i>NaCuSO₄</i>			
Na ₁	0.00000	0.50000	0.50000
Cu ₂	0.25000	0.25000	0.00000
S ₃	0.09090	0.00000	0.30130
O ₄	0.19310	0.00000	0.19190
O ₅	0.20910	0.00000	0.51770
O ₆	− 0.02010	0.19670	0.24400
O ₇	0.15760	0.50000	0.07540
H ₈	0.01690	0.50000	0.00690
H ₉	0.20700	0.50000	0.22390

and NaCuSO₄ nanoparticles are shown in Fig. 5a–d. The average grain size of the CuO nanoparticles was calculated from the three most intense peaks using Debye–Scherrer's formula. The resultant average grain size of CuO nanoparticles was calculated from the Debye–Scherrer formula as 40 nm.

W–H plot analysis

The Williamson–Hall (W–H) plot method is used to calculate the crystallinity size and strain of the bio synthesized CuO nanoparticles from the XRD data. The incoherent diffraction domains induce the micro-strains in the CuO nanoparticles. The experimental data of the XRD pattern shows the appearance of peak broadening, where the broad peaks indicate the smaller crystal size of the nanoparticles (Ravichandran et al. 2022). Let the broadening of peak β_T , crystallite size β_D and micro-strain β_ϵ are related by the equation,

Total broadening = Broadening due to (crystallite size + strain).

$$\beta_T = \beta_\epsilon + \beta_D \quad (3)$$

Table 3 Lattice parameters of CuO and NaCuSO₄ nanocomposites

Formula	Hall Symbol	a (Å)	α (degree)	b (Å)	β (degree)	c (Å)	γ (degree)
CuO	C -2yc	4.68318	90°	3.42184	99.49068°	5.12653	90°
NaCuSO ₄	-C 2y	8.82040	90°	6.19115	118.62009°	7.50736	90°

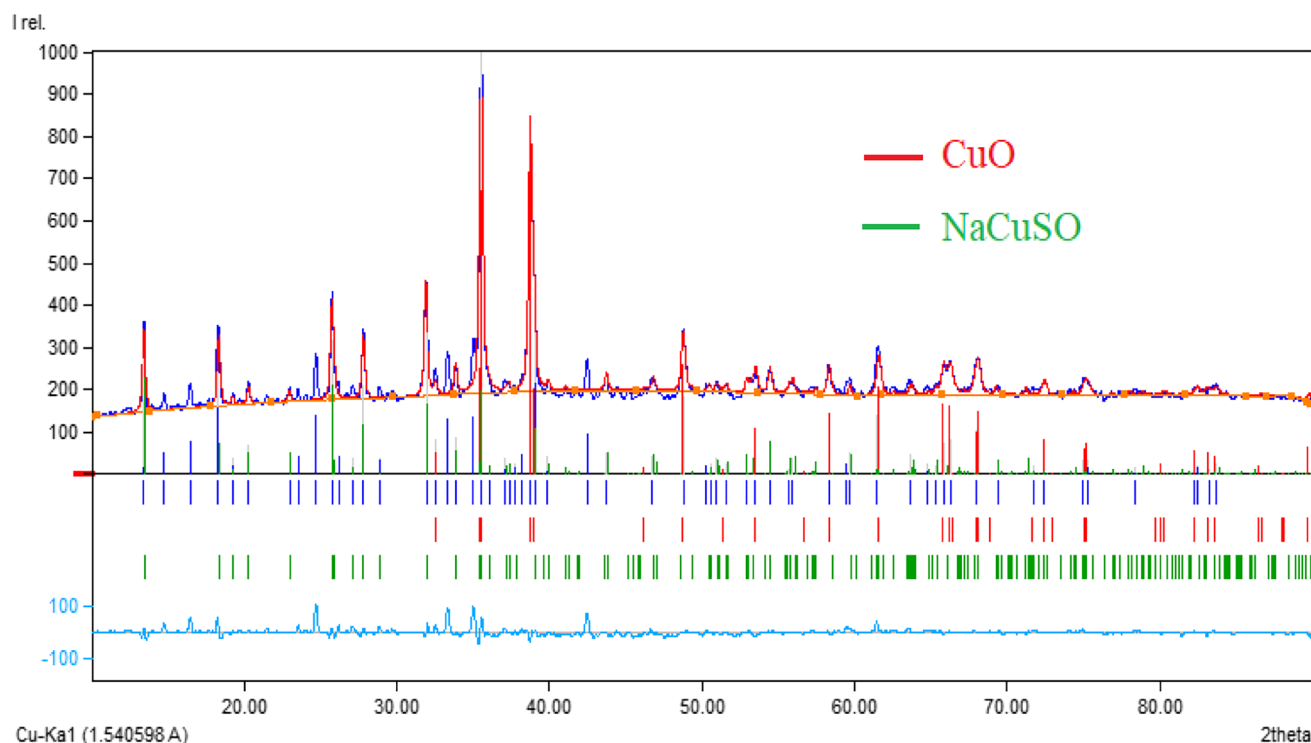


Fig. 4 Rietveld refined XRD pattern of CuO/NaCuSO₄ nanocomposite

The micro-strain can be calculated by using the relation.

$$\beta_{\epsilon} = 4\epsilon \tan \theta \quad (4)$$

From the Scherrer equation,

$$\beta_D = \frac{K\lambda}{D \cos \theta} \quad (5)$$

Substituting Eqs. (4) and (5) in Eq. (3), we get

$$\beta_T = 4\epsilon \tan \theta + \frac{k\lambda}{D \cos \theta} \quad (6)$$

Simplifying and get,

$$\beta_T \cos \theta = 4\epsilon \sin \theta + \frac{k\lambda}{D} \quad (7)$$

Equation (7) indicates a straight-line equation in which the slope of the linear fit is ϵ (strain) and the y-intercept ($\frac{k\lambda}{D}$) is used to calculate crystalline size. W–H plot can be studied by plotting the graph between $4\epsilon \sin \theta$ and the y-axis $\beta_T \cos \theta$ (Harikrishnan et al. 2022). The nanocrystalline size and microstrain were calculated from the graph shown in Fig. 6. The value of micro-strain is 7.75×10^{-4} and the crystalline size is 38.84 nm.

SEM analysis & EDX analysis

The scanning electron microscope is a potent analytical tool that can produce high-resolution images and perform analysis on a variety of substances at high magnifications. A scanning electron microscope (SEM) scans using a focused electron beam over a surface to create an image. The electrons in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and composition. The detection of high-energy electrons emitted from the surface of a sample after it has been exposed to a highly focused beam of electrons from an electron cannon is the basis of scanning electron microscopy. Using the SEM objective lens, this electron beam is focused on a small spot on the sample surface. There are two modalities of electron detection, which allow for various types of imaging and analysis. Backscattered electrons can be detected to provide contrast based on differences in chemical composition throughout an image. Secondary electrons emitted near the surface of the sample provide information about its topography. The morphological studies were done by synthesized CuO nanoparticles, which clearly show the morphology of the composite. The precursors are responsible for the different morphologies of the *Tribulus*

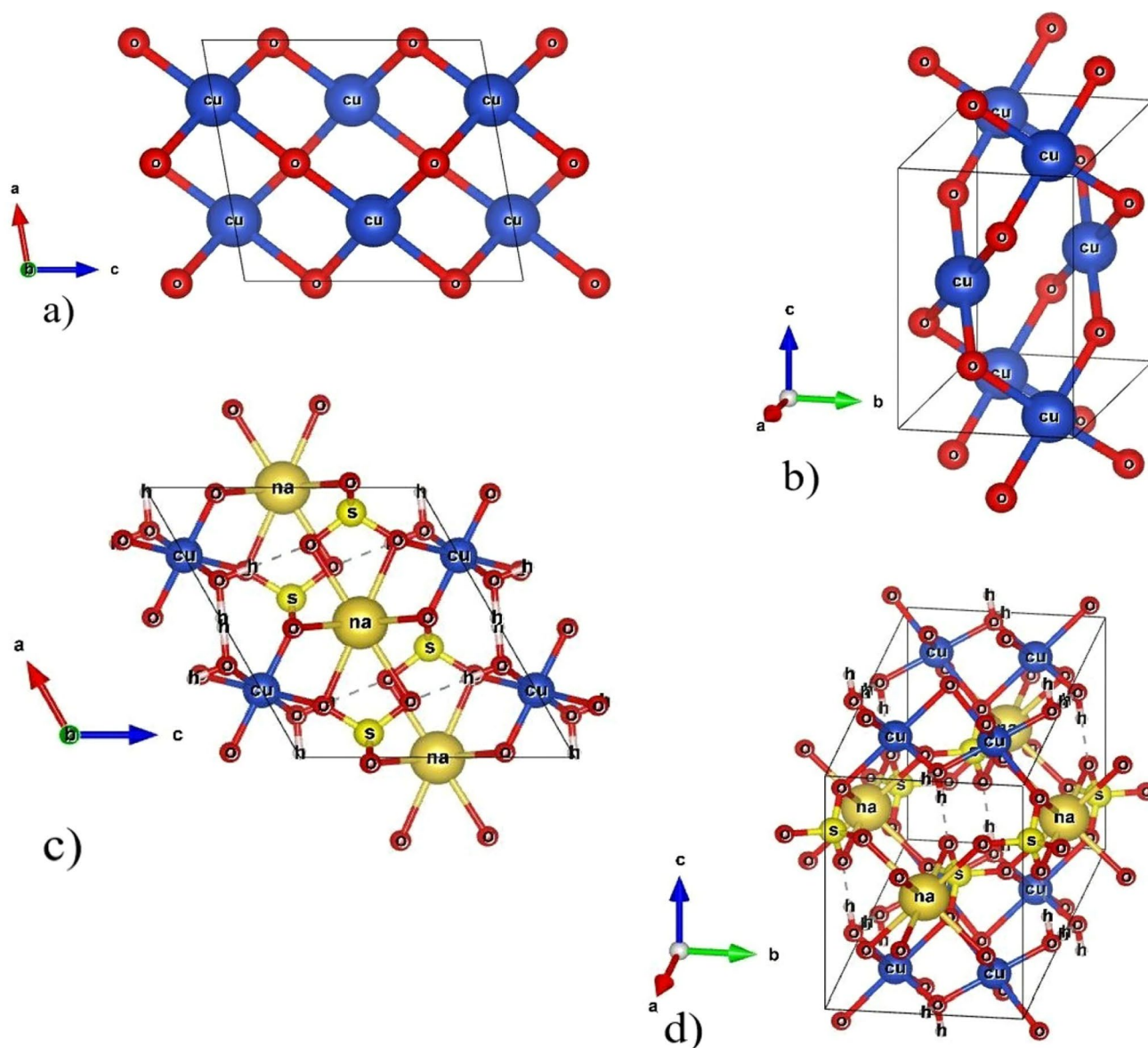


Fig. 5 a–b 2-D and 3-dimensional view of biosynthesized CuO nanoparticles; c–d 2-D and 3-dimensional view of NaCuSO₄ nanoparticles

terrestris—mediated CuO nanomaterial. The CuO nanoparticles were imaged with CAREL ZEISS microscopy EVO 18 model, which magnifies the sample with different magnification ranges. The magnification range at M_{ag} = 45.00, 40.00, 35.00, 25.00, 20.00, and 15.00 KX as shown in Fig. 7a–f and it clearly shows that the copper sulfate was utilized as a precursor, the copper oxide molecules grown slowly and form cluster-like CuO nanostructures which were observed at the different magnification range. The CuO nanoclusters have a size range of 103 nm to 159 nm. Therefore, *Tribulus terrestris* acts as a capping agent, it pursues the variations of morphologies in CuO nanoparticles. The elemental identification was done by energy dispersive spectrophotometer. The observed results are affirming to the obtained results of

XRD analysis. The identification of elemental composition is illustrated in Fig. 8, which clearly shows the presence of copper, oxygen, sodium, and sulfur in the mass percentage of 52.7%, 37.2%, 1.4% and 8.6%, respectively. Which confirms the formation of CuO and NaCuSO₄ nanocomposites, respectively.

Dynamic light scattering analysis

The characterization of particle sizes in suspensions and emulsions is done using dynamic light scattering, a reliable and accurate measurement method. For over 30 years, optical systems based on dynamic light scattering have been developed by microtrac, a pioneer in the field of particle analysis

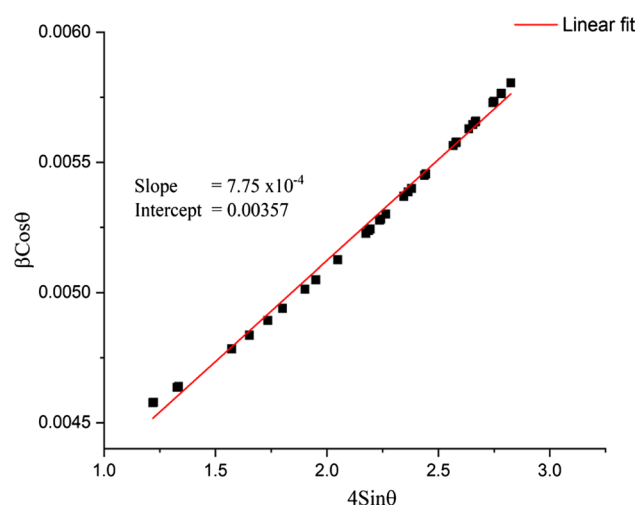


Fig. 6 W–H plot analysis of CuO nanoparticles

technology. The dynamic light scattering analysis (DLS) technique was used to study the particle size of the CuO nanoparticles. Bio reduction of *Tribulus terrestris* aqueous extract with CuO nanoparticles is also by the particle size investigation method. In Fig. 9, the size distribution of the synthesized composite was measured from the ranges 20 nm–112 nm. The particle size measured by this scattering technique is marginally greater than the size measured by SEM micrographs. Because the hydrodynamic radius method was used to calculate the particle size.

Dielectric studies

Dielectric materials are categorized as insulating materials or poor conductors. Using the dielectric spectroscopy technique, we can examine the characteristics of dielectric materials including their dielectric loss and dielectric constant. The sample material is placed between two electrodes, and it is initially exposed to alternating electric field frequencies. The output is monitored on the frequency being used from the applied frequency. This frequency belongs to the microwave or radio spectrum. These fields induce polarization which causes dipoles to shift either translationally or rotationally. Electromagnetic radiation is used as a source for a variety of optical characterization techniques including Raman, IR, and fluorescence analysis. However, in broadband spectroscopy alternating external voltage is used as a source to produce various kinds of polarization.

The following relation gives the relationship between ϵ' , C , d , and ϵ_0 (Cardarelli et al. 2008).

$$\epsilon' = \frac{Cd}{\epsilon_0} \quad (8)$$

where ϵ_0 , C and ' d ' represents the permittivity of free space, observed capacitance and thickness of the nano-composite pellet materials, respectively.

$$\tan\delta = \frac{\epsilon''}{\epsilon'} = \frac{1}{RC\omega} \quad (9)$$

where ϵ' , ϵ'' , R , ω , and $\tan\delta$ denote the real part of dielectric constant, imaginary part of the dielectric constant, resistance, angular frequency, and dielectric loss, respectively. The electrical field activates freely coupled molecules into polarized state, in contrast the carrier ions will be moved away from the equilibrium state. As a result, polarization processes, the charge separations in dielectric materials allow for the flow of current that is produced by the electric field components. Dielectric materials are generally considered to have low electrical conductivity and have the capacity to hold or store the electrical charges because of the process due to the action of dielectric polarization on the material. Hence these types of materials are called dielectric materials. The term dielectric constant is used to determine how well a substance can hold electrical energy in the presence of alternating electric field frequencies and is called dielectric constant. Normally the relative permittivity is a medium of dielectric constant, i.e., the ratio of relative permittivity of the medium with respect to the permittivity of free space (Cardarelli et al. 2008). The dielectric constant of nanocomposites can be derived with the help of broadband spectrometer. From the observed values, we plot a graph as shown in Fig. 10a on the dielectric constant by the applied frequency with respect to the real and imaginary parts of the dielectric constant. That is permittivity of a medium and free space as shown in Fig. 10a green color sphere dots and red color square dots referred as a real and imaginary part of the dielectric constant (ϵ' and ϵ''). At low frequencies, the dielectric constant of a real part and imaginary part was very high. At low frequencies, the real and imaginary parts of the dielectric constant gradually decreased (Izgorodina et al. 2009). There are some types of polarization (orientation, electronic and ionic) that can be used to explain how the dielectric constant decreases with applied frequency. When an electric field is applied to a material, the ionic polarization results in the positive ions moving away from the negative ions. Orientation, polarization is attributed to the material's structure, that emerges according to the frequency of 1 kHz to 1 MHz (Bouaamlat et al. 2020). . Electronic polarization occurs when the atom's electron cloud is moved away from the nucleus. With regard to the final one, electron polarization arises from the nucleus of the atom being physically displaced with respect to all of the electrons present in it. This kind of polarization can be identified relatively very little bit time response and

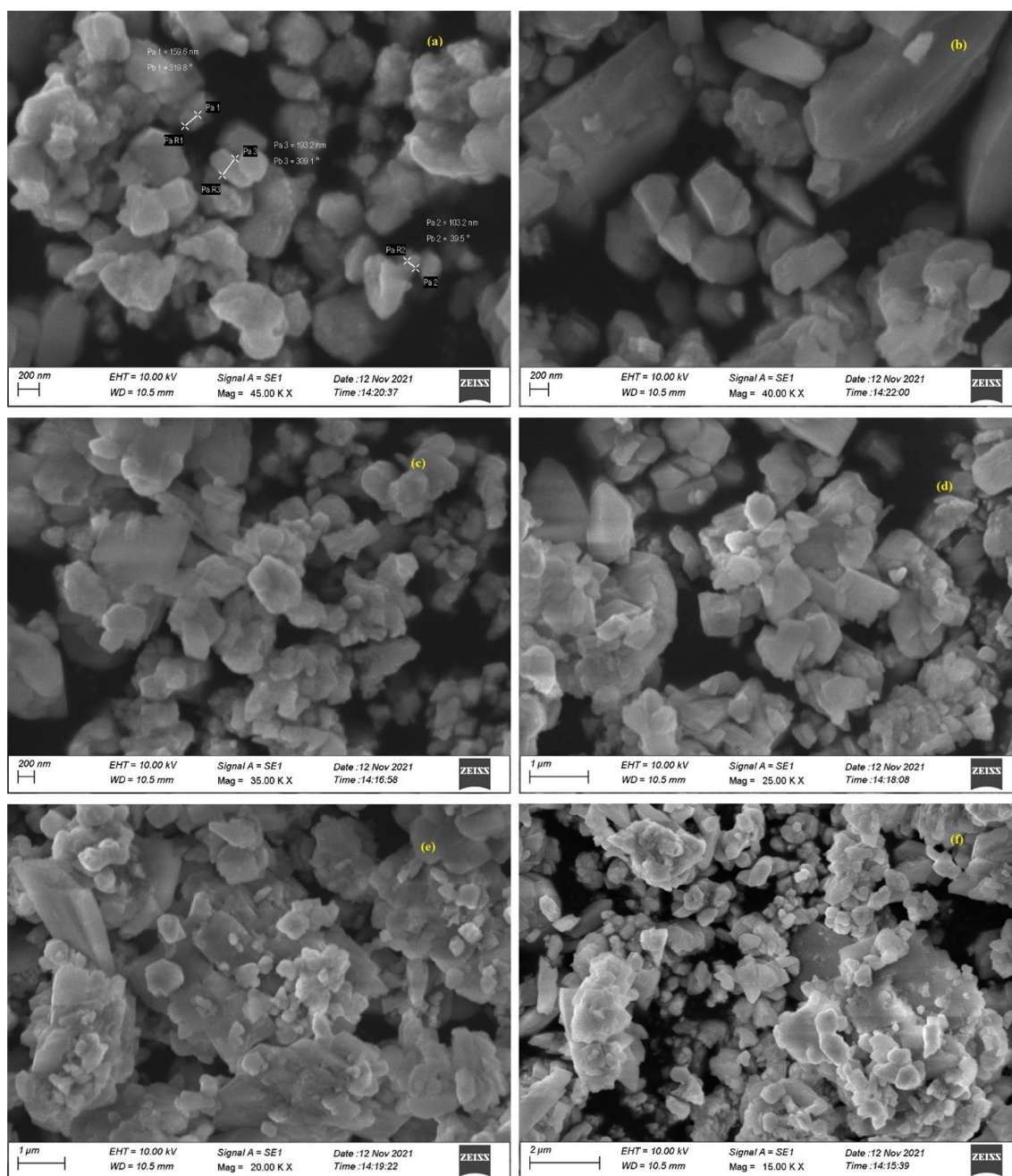


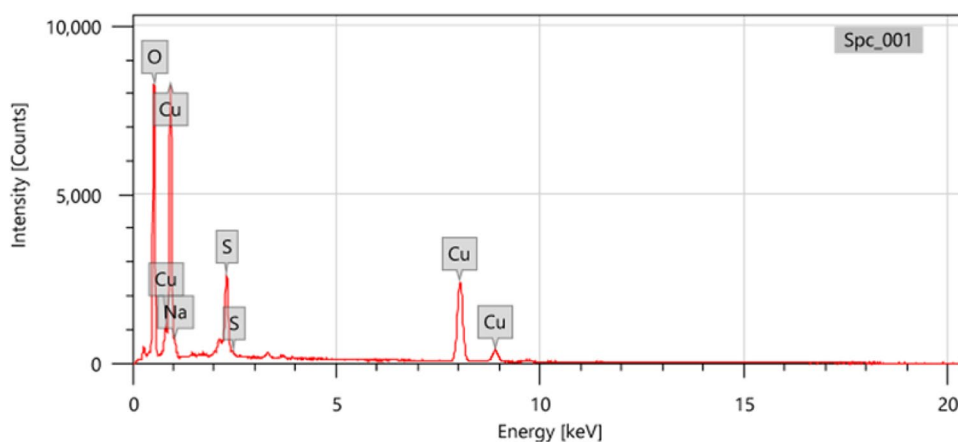
Fig. 7 a–f. SEM analysis of CuO nanoparticles

retains its sensitivity up to the frequencies higher than the frequency of visible light. The orientation polarization is more common due to its longer development time compare than other polarizations, as a result, the decreasing of dielectric constant is reached a constant value at high frequency, which pertains to interfacial polarization. Since the CuO nanomaterial has higher dielectric constant with lower frequency which means it has high storing capacity.

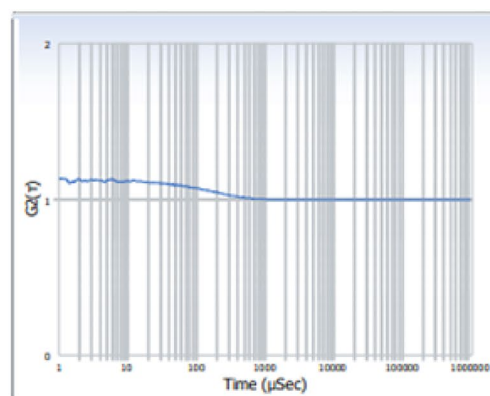
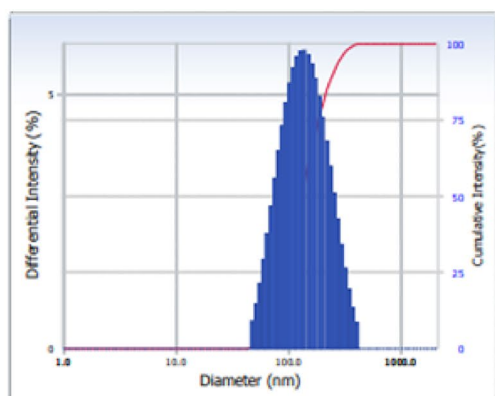
The dielectric loss is the amount of energy consumed during the heat treatment of a dielectric substance in a variety

of electric domains. As an illustration, a capacitor integrated into the circuit of an alternating current barely receives a charge or discharge of every half cycle. The dielectric factor measures how much energy of the applied frequencies can absorb into the medium. Most significantly, a dielectric loss often depends on the applied frequency and the dielectric substance. The loss tangent or $\tan \delta$ is used to evaluate the dielectric loss. The movement of ions transfers some of their energy to the crystal lattice, hence the electrical energy dissipates as heat (Hameed et al. 2022).

Fig. 8 Elemental analysis spectrum of biosynthesized CuO & NaCuSO₄ nanocomposites



Element	Line	Mass%	Atom%
O	K	37.21±0.17	66.69±0.30
Na	K	1.44±0.09	1.79±0.11
S	K	8.65±0.08	7.73±0.07
Cu	K	52.71±0.35	23.78±0.16
Total		100.00	100.00
Spc_001			Fitting ratio 0.0319



Distribution Results (Contin)

Peak	Diameter (nm)	Std. Dev.
1	149.7	71.8
2	0.0	0.0
3	0.0	0.0
4	0.0	0.0
5	0.0	0.0
Average	149.7	71.8
Residual :	2.634e-003	(O.K)

Cumulants Results

Diameter (d)	: 115.7	(nm)
Polydispersity Index (P.I.)	: 0.223	
Diffusion Const. (D)	: 4.251e-008	(cm ² /sec)
Measurement Condition		
Temperature	: 25.0	(°C)
Diluent Name	: WATER	
Refractive Index	: 1.3328	
Viscosity	: 0.8878	(cP)
Scattering Intensity	: 28882	(cps)
Attenuator 1	: 1.56	(%)

Fig. 9 Dynamic light scattering analysis of CuO nanoparticles

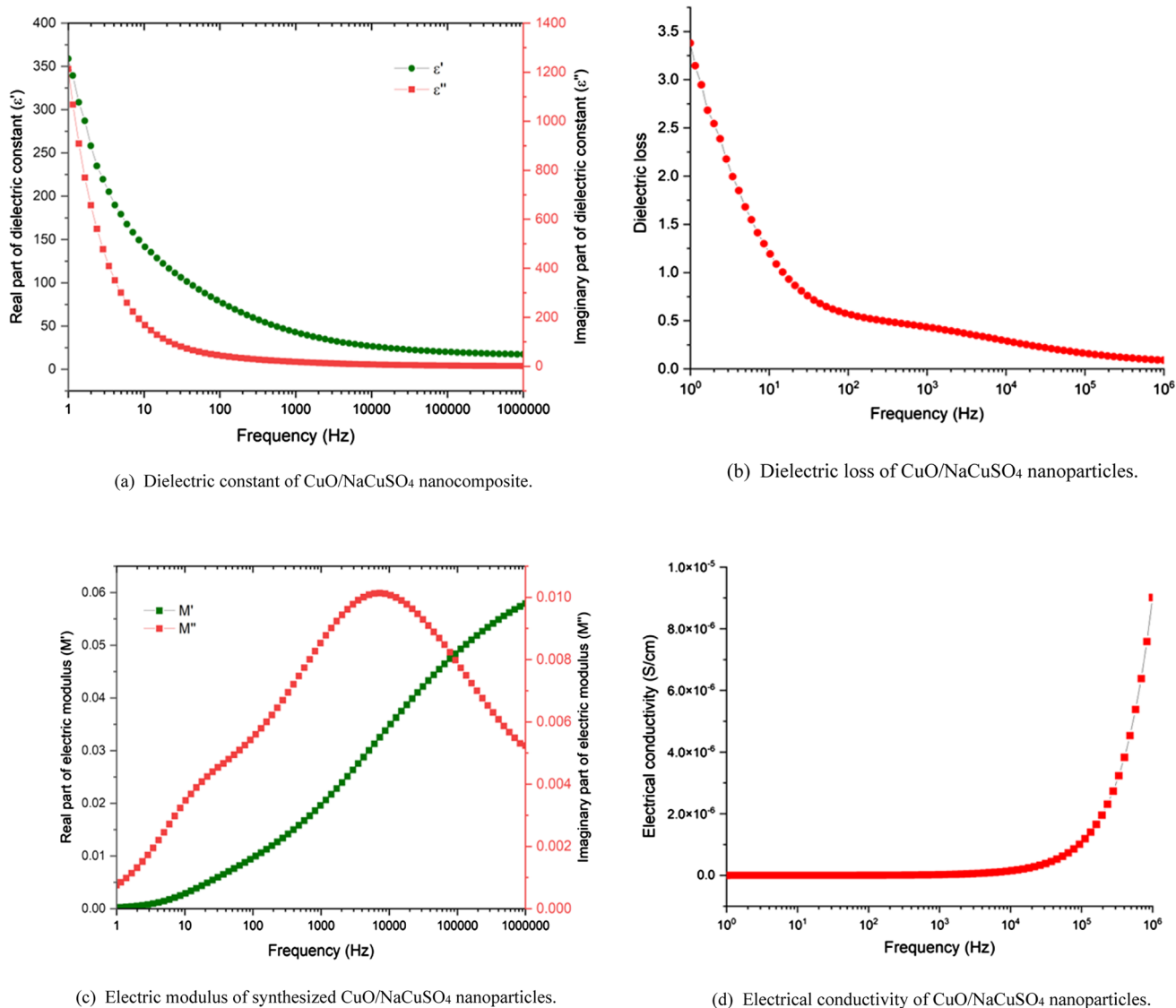


Fig. 10 **a** Dielectric constant of CuO/NaCuSO₄ nanocomposite. **b** Dielectric loss of CuO/NaCuSO₄ nanoparticles. **c** Electric modulus of synthesized CuO/NaCuSO₄ nanoparticles. **d** Electrical conductivity of CuO/NaCuSO₄ nanoparticles

From graphical analysis report as shown in Fig. 10b, it was found that the nanocomposite exhibits higher dielectric loss and at low frequencies, material shows very lower amount of dielectric loss. Which is nearly equal to zero, it means our synthesized nanocomposite has very low electrical loss. Hence, it may be used to store energy in the place of energy storing capacitors. The complex electric modulus is a highly effective tool for examining the dielectric behavior of an insulating material. It is the inverse form of complex relative permittivity (Hodge et al. 2005). From the graph as shown in Fig. 10c the green and red colored lines indicate the real and imaginary parts of the electric modulus with respect to the applied field frequency. At low frequency, the real part of the complex electric modulus (M') is negligible. As increasing the applied frequency, the electric modulus

also increased gradually. By the way, the imaginary part of the electric modulus also increased followed by the applied frequency.

Electrical conductivity is a measurement of a material's electrical current carrying capacity or conductivity. The term specific conductance also refers to electrical conductivity (El-Mallah et al. 2012). How readily a material permits electric current to pass through it is determined by its electrical conductivity. On the other hand, the electrical resistivity predicts how strongly a substance is opposed by the flow of electric current. Materials conductivity can be affected by the temperature in a less obvious way. An increase in temperature typically reduces the conductivity of materials such as conductors. But in the case of insulators, the situation is different as rising the temperature makes them

more conductive. The electrical conductivity of composite is studied and shown in Fig. 10d. From the graphical analysis, the synthesized CuO nanoparticles have constant electrical conductivity behavior up to 10^5 Hz and after crossing this frequency, the conductivity of a CuO material suddenly increased at a higher rate. It shows well a substance can carry electrical current. The dielectric conductivity adds together all dissipative effects, which could be the conductivity brought on by moving charge carriers and connected to a frequency dependent energy loss.

Conclusion

In this report, we have investigated the interaction between an eco-friendly species of *Tribulus terrestris* aqueous extract and the progenitor salt of copper sulfate followed by green synthesis method. The formation and stabilization of the CuO/NaCuSO₄ nanocomposite have also been examined. Nanocomposites were successfully synthesized and analyzed by different characterization techniques such as XRD, UV–vis, SEM, DLS, and FTIR analysis. From the UV–vis spectroscopic technique, the energy bandgap was calculated as 3.6 eV, which was observed from the maximum absorption peak wavelength at 337 nm. The functional information analysis was done by Fourier transform infrared spectroscopy, which clearly shows the formation of CuO nanoparticles. In the metal oxide region, the CuO nanoparticles were confirmed at 526 cm^{-1} . From the powder X-ray diffraction analysis technique, it was found that the CuO and NaCuSO₄ nanocomposites have monoclinic crystal structures, and their crystal size was calculated as 40 nm (using Debye Scherrer method), respectively. The scanning electron microscopy analysis was done on the green synthesized CuO/NaCuSO₄ nanoparticles. The cube-like nanocrystals were observed, with a size range of around 100 nm. From the EDAX analysis, we confirmed the presence of elements with their mass percentage namely Cu (52.7%), O (37.2%), S (8.6) and Na (1.44%). The broadband dielectric analysis revealed the dielectric behavior of CuO nanoparticles. The parameters of dielectric constant, dielectric loss, electrical modulus, and electrical conductivity have also been analyzed. The electrical modulus was found negligible at lower frequencies, and higher electrical conductivity was observed at higher frequencies.

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