



# Biofabricated CuO Nanoparticle Reinforced Bamboo Fiber Coatings for Photocatalytic Antimicrobial and Superhydrophobic Biomedical Textile Applications

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## ABSTRACT

Controlling the physicochemical and functional properties of green-synthesized CuO nanoparticles remains a significant challenge for antimicrobial textile applications. This study investigated the influence of wet-chemical, hydrothermal, and sonication-assisted synthesis methods on the performance of CuO nanoparticles prepared using *Hibiscus rosa-sinensis* leaf extract. Among the investigated methods, hydrothermal synthesis produced CuO nanoparticles with enhanced crystallinity and an average crystallite size of 54.2 nm. These nanoparticles exhibited superior photocatalytic degradation efficiency (~95%), stronger antibacterial activity against *Staphylococcus aureus* (21.1 mm) and *Escherichia coli* (21.0 mm), and lower defect density compared with those synthesized by other methods. The optimized nanoparticles were incorporated into a chitosan coating and applied to bamboo fabric. The coated fabrics demonstrated superhydrophobic behavior with a water contact angle of  $157 \pm 0.1^\circ$  and excellent antibacterial performance, achieving inhibition zones of  $21 \pm 1.1$  mm (~96%) against *E. coli* and  $30.1 \pm 1.1$  mm (~99%) against *S. aureus*. The enhanced performance was attributed to improved crystallinity, reduced defect density, favorable surface morphology, and effective nanoparticle dispersion. The study demonstrates the significant influence of synthesis conditions on CuO nanoparticle performance and highlights their potential for developing antimicrobial and water-repellent bamboo-based textiles for biomedical and protective applications.

**Keywords:** Green nanotechnology; CuO nanostructures; Plant-mediated synthesis; Functional textiles; Water-repellent coating; Bacterial inhibition.

## 1. INTRODUCTION

The growing demand for sustainable and environmentally friendly nanomaterials has stimulated significant interest in green synthesis approaches utilizing plant-derived extracts as reducing and stabilizing agents. Compared with conventional chemical methods, plant-mediated synthesis offers advantages such as reduced toxicity, environmental compatibility, cost-effectiveness, and the utilization of naturally occurring phytochemicals. Among various medicinal plants, *Hibiscus rosa-sinensis* has emerged as a promising bioresource because of its rich content of phenolics, flavonoids, tannins, and carotenoids, which facilitate nanoparticle formation and stabilization.

Several studies have demonstrated the successful synthesis of functional nanomaterials using *Hibiscus rosa-sinensis* extracts. Green-synthesized CuO nanoparticles exhibited effective antibacterial activity and photocatalytic degradation performance

(Velayutham *et al.* 2023). Similarly, Hibiscus-mediated ZnO nanostructures showed enhanced antimicrobial properties and controlled morphology suitable for biomedical applications (Elemike *et al.* 2021). Biosynthesized  $\text{CoFe}_2\text{O}_4$  nanoparticles demonstrated promising antimicrobial and pollutant degradation capabilities (Velayutham *et al.* 2022), while CuI and Ag nanoparticles synthesized using *Hibiscus rosa-sinensis* extracts exhibited strong antibacterial activity against pathogenic microorganisms (Archana, *et al.* 2021b). Green-synthesized CuO nanoparticles derived from *Ocimum lamiifolium* and *Withania somnifera* leaf extracts exhibited crystallite sizes of 15–19 nm and significant antibacterial activity against *S. aureus*, *E. coli*, and *P. aeruginosa* (Mengesha *et al.* 2024). Furthermore, CuO nanoparticles synthesized using *Citrus aurantium* peel extract achieved methylene blue degradation efficiencies of 95.34–97.5% and exhibited particle sizes ranging from 53.25 to 68.02 nm, demonstrating their potential for environmental remediation applications (Khedr and Ali, 2024). Although these studies

demonstrate the versatility of *Hibiscus rosa-sinensis* extracts for synthesizing various nanomaterials, most investigations primarily focus on nanoparticle preparation and biological activity, whereas comparative assessments of synthesis routes and their influence on multifunctional applications remain limited (Munir *et al.* 2025).

Among various metal oxide nanomaterials, copper oxide (CuO) nanoparticles have attracted considerable attention owing to their excellent photocatalytic activity, broad-spectrum antibacterial efficacy, chemical stability, and low production cost. These characteristics have enabled their application in environmental remediation, antimicrobial coatings, wastewater treatment, and biomedical materials. Previous investigations reported that CuO/TiO<sub>2</sub> nanocomposites achieved high photocatalytic degradation efficiencies for organic pollutants (Nguyen *et al.* 2024), while mesoporous CuO-based systems demonstrated effective adsorption and removal of dye contaminants from aqueous environments (Nain *et al.* 2025). Furthermore, CuO-containing adsorbents have exhibited excellent reusability and pollutant removal performance, highlighting their multifunctional potential in environmental applications (Umeh *et al.* 2025).

The properties of CuO nanoparticles are strongly influenced by the synthesis route, which affects crystallinity, particle size, morphology, defect density, and consequently their photocatalytic and antibacterial performance. Although *Hibiscus rosa-sinensis*-mediated synthesis of CuO nanoparticles has been previously reported, available studies have largely focused on nanoparticle preparation and antimicrobial evaluation. As a result, comparative investigations of wet-chemical, hydrothermal, and sonication-assisted synthesis routes using *Hibiscus rosa-sinensis* extract remain limited, hindering the optimization of nanoparticle functionality. The multifunctional properties of CuO nanoparticles make them attractive candidates for incorporation into textile substrates, where they can impart antimicrobial, photocatalytic, and protective functionalities. Alongside advances in nanoparticle synthesis, considerable attention has been devoted to multifunctional textile materials. Bamboo fabric is a sustainable textile substrate owing to its biodegradability, renewability, and comfort properties; however, its inherent antibacterial and water-repellent characteristics are limited. Incorporating metal oxide nanoparticles into textile coatings offers an effective approach for imparting antimicrobial, self-cleaning, and hydrophobic functionalities.

Previous studies have demonstrated that nanoparticle-modified bamboo-based materials exhibit enhanced performance. ZnO-loaded bamboo fiber composites showed improved antibacterial activity and structural properties (Liu *et al.* 2024), while functionalized bamboo composites displayed enhanced

microbial resistance and durability (Szadkowski *et al.* 2024). Similarly, modified bamboo films with improved nanoparticle loading capacity demonstrated superior antibacterial characteristics (Zhan *et al.* 2026). Antibacterial treatments applied to bamboo-derived materials have further confirmed their potential for protective textile applications (Zhang *et al.* 2021). These findings suggest that bamboo fabrics can serve as effective substrates for multifunctional nanocomposite coatings.

Despite these developments, a systematic correlation between the synthesis route of *Hibiscus rosa-sinensis*-derived CuO nanoparticles and their photocatalytic, antibacterial, and textile functionalization performance has not been comprehensively established. To the best of our knowledge, no previous study has comparatively evaluated wet-chemical, hydrothermal, and sonication-assisted *Hibiscus rosa-sinensis*-mediated CuO nanoparticles and correlated their properties with the performance of CuO-coated bamboo fabrics. Moreover, the influence of wet-chemical, hydrothermal, and sonication-assisted synthesis methods on the multifunctional properties of CuO-coated bamboo fabrics remains largely unexplored.

Therefore, the present study comparatively investigates the synthesis of CuO nanoparticles using *Hibiscus rosa-sinensis* leaf extract through wet-chemical, hydrothermal, and sonication-assisted routes. The synthesized nanoparticles were evaluated in terms of their structural, optical, photocatalytic, and antibacterial properties, and the optimum nanoparticles were subsequently incorporated into a chitosan matrix for bamboo fabric coating. This work provides valuable insight into the relationship between synthesis conditions and nanoparticle functionality while demonstrating a sustainable approach for developing antimicrobial and water-repellent bamboo-based textiles for advanced biomedical and protective applications.

## 2. MATERIALS AND METHODOLOGY

### 2.1 Materials

Fresh *Hibiscus rosa-sinensis* leaves were collected from Coimbatore, Tamil Nadu, India, washed thoroughly with double-distilled water, and shade-dried for two weeks. The dried leaves were ground using a ball mill for 15 h to obtain a fine powder. To prepare the extract, 10 g of powdered leaf material was suspended in 100 mL of double-distilled water and permitted to stand for 24 hours to promote phytochemical extraction. Mixture were filtered using filter paper to obtain a clear aqueous leaf extract, which served as a stabilizing and reducing medium for green production of CuO NPs. Copper nitrate trihydrate [Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O, ≥99%, Sigma-Aldrich], sodium hydroxide (NaOH, ≥98%, Merck), ethanol (99.9%, Merck), glacial acetic acid

(99.8%, Merck), and chitosan (medium molecular weight, Sigma-Aldrich) were used without further purification.

## 2.2 Synthesis of CuO NPs

In nanoparticle synthesis, a suitable concentration of copper precursor solution was incrementally added to the extract while maintaining continuous stirring, resulting in the creation of CuO NPs through phytochemical-mediated reduction (Fardood *et al.* 2026). Copper nitrate trihydrate  $[\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}]$  was used as the copper precursor at a concentration of 0.1 M. The pH of the reaction mixture was adjusted to 10 using 1 M NaOH solution and maintained throughout the synthesis process. Following nanoparticle formation, the precipitate was separated by centrifugation at 6000 rpm for 15 min, washed repeatedly with distilled water and

ethanol, and subsequently dried. Calcination was performed at 600 °C for 2 h with a heating rate of 10 °C  $\text{min}^{-1}$  to obtain crystalline CuO nanoparticles. The reaction mixture was kept at regulated pH and temperature levels until a discernible color change indicated nanoparticle production. The precipitate was recovered via centrifugation, thoroughly washed with distilled water and ethanol to remove residual contaminants, and dried and calcined at high temperature to produce crystalline CuO nanoparticles with improved purity and stability. During the green synthesis of CuO nanoparticles, the phytochemical constituents present in *Hibiscus rosa-sinensis* extract function as reducing, stabilizing, and capping agents, as illustrated in Figure 1. The common post-synthesis steps, including centrifugation, washing, drying, and calcination, were applied to all three synthesis routes unless otherwise specified.

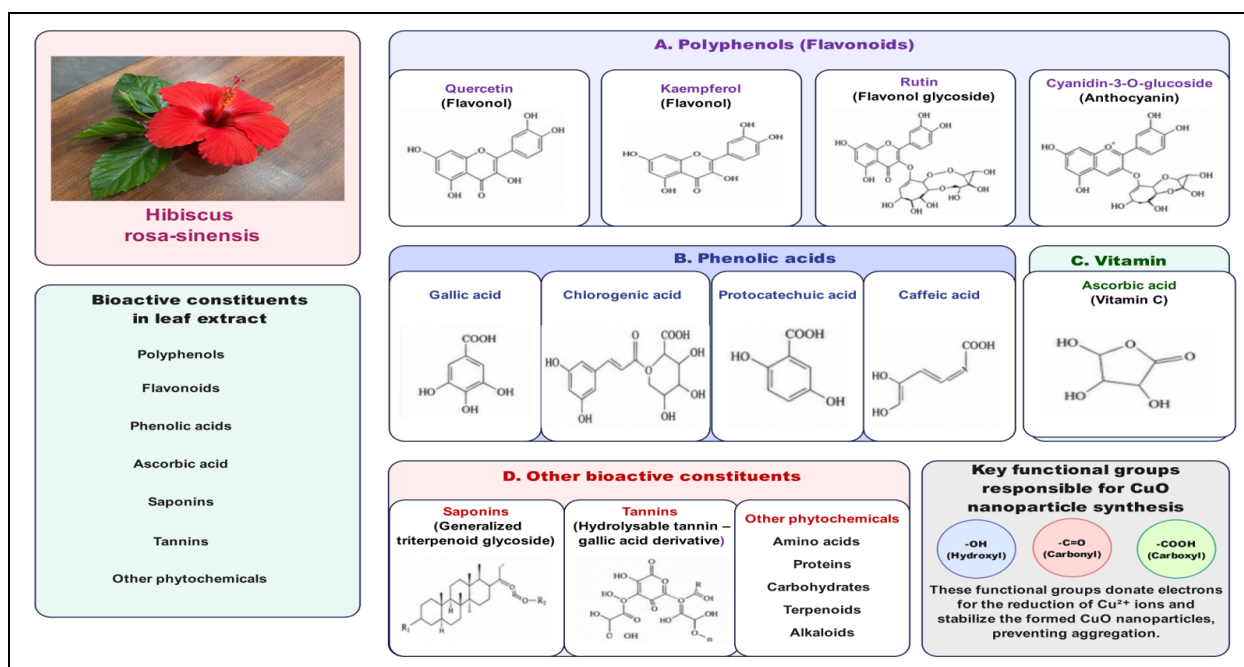


**Fig. 1: Major phytochemical constituents present in *Hibiscus rosa-sinensis* leaf extract responsible for the reduction, stabilization, and capping of CuO nanoparticles during green synthesis**

### 2.2.1 Sonication

The precursor solution was sonicated at 40 kHz for 1 h to ensure homogeneous dispersion (Archana *et al.* 2022). The synthesized nanoparticles produced by this method were labeled as CuO-s.

### 2.2.2 Wet-chemical

In the wet-chemical synthesis of CuO nanoparticles, the precursor solution was agitated continuously with a magnetic stirrer at an elevated temperature of 70 °C for 1 hour until visible precipitates formed. The nanoparticles generated through this process were labeled as CuO-w.

### 2.2.3 Hydrothermal

The precursor solution was transferred to a Teflon-lined autoclave and maintained at 220 °C for 12 h under autogenous pressure to facilitate crystal growth and nanoparticle formation. Such circumstances generally enhance crystallinity while diminishing defect density relative to traditional high-temperature methods. The synthesized nanoparticles were labeled as CuO-h.

## 2.3 Characterization of CuO NPs

X-ray diffraction (XRD) analysis was performed to determine the crystalline phase and structural characteristics of CuO-s, CuO-w, and CuO-h

samples. The diffraction patterns were obtained utilizing a Phillips X'Pert MPD powder diffractometer with Cu-K $\alpha$  radiation operating at 30 mA and 40 kV in Bragg–Brentano geometry. Data capture occurred within the 2 $\theta$  range of 10°–80° with a step size of 0.02°, facilitating comprehensive assessment of phase composition and crystallinity of the synthesized samples.

Fourier transform infrared (FTIR) spectroscopy was employed to identify the functional groups associated with the synthesized samples, employing a Spectrum 100 spectrometer across a wavenumber range of 4000–400 cm<sup>-1</sup>. The samples were generated by dispersing CuO nanoparticles in potassium bromide (KBr) at a constant mass ratio of 1:200, followed by pellet production using a hydraulic press to ensure uniform mixing and accurate spectrum collection.

The KBr–CuO pellet was analyzed to acquire infrared spectra for functional group identification. Particle size distribution was measured using a Nanophox analyzer (Sympatec, Germany) based on dynamic light scattering principles. Surface morphology and elemental composition were evaluated using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM–EDX).

The optical properties of the produced CuO nanoparticles were examined by a UV–Vis spectrophotometer (Cary 8454, Agilent, Singapore)

across a wavelength range of 200–800 nm with suitable spectral resolution. Photoluminescence (PL) measurements were conducted utilizing a fluorescence spectrophotometer to investigate emission characteristics. The absorption and emission profiles were rigorously assessed to elucidate the excitonic transitions, charge carrier dynamics, and comparative optical characteristics of the CuO nanoparticle systems.

## 2.4 Photocatalytic Activity

Photocatalytic efficacy of green-synthesized CuO NPs (CuO-s, CuO-w, and CuO-h) obtained from *Hibiscus rosa-sinensis* leaf extract was assessed via degradation of methylene blue (HiMedia, Mumbai, India) at UV irradiation. Prior to UV irradiation, the suspension containing 100 mg mL<sup>-1</sup> methylene blue and 1 g L<sup>-1</sup> catalyst was magnetically stirred in the dark for 30 min to establish adsorption–desorption equilibrium. Control experiments were conducted in the absence of catalyst and UV irradiation. The UV intensity at 365 nm was maintained throughout the experiment. The studies were performed in a cubic UV reactor fitted with a 40 W annular lamp ( $\lambda = 365$  nm). Aqueous dye solutions with dispersed CuO nanoparticles were continually agitated using a magnetic stirrer to maintain uniform suspension and facilitate effective interaction during irradiation. The experimental setup employed for photocatalytic degradation studies is illustrated in Fig. 2.

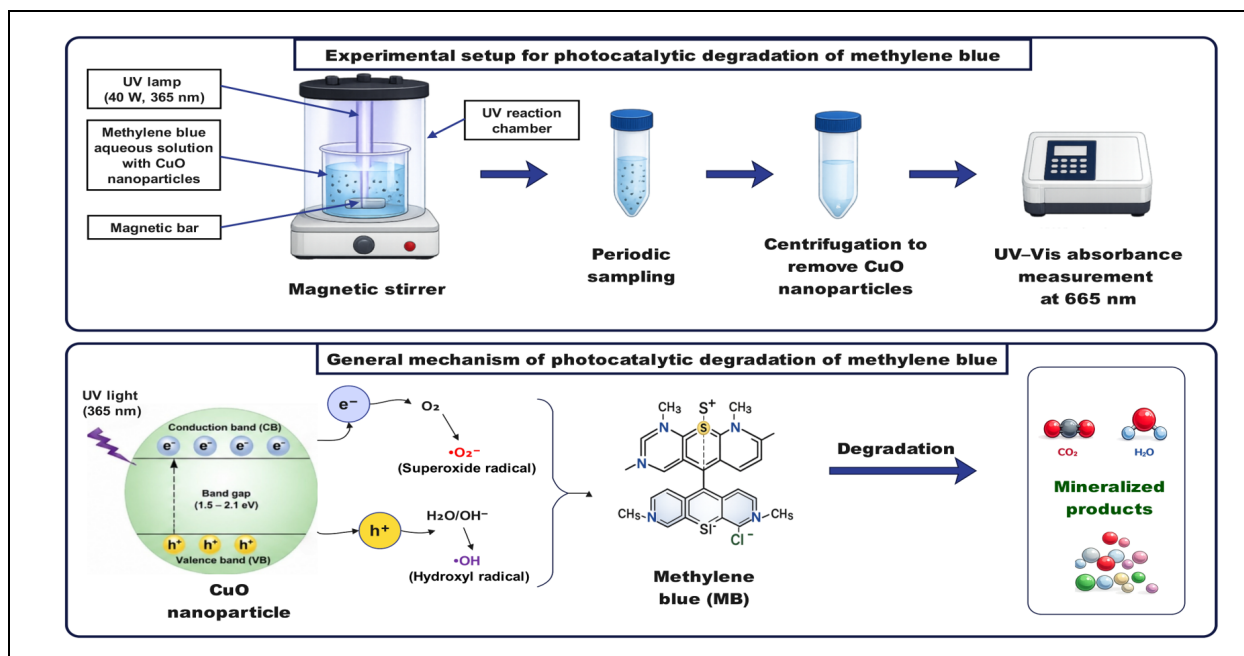


**Fig. 2: Experimental setup for photocatalytic degradation of methylene blue using CuO nanoparticles**

During the photocatalytic assessment, 10 mL aliquots were periodically extracted from the reaction system at 30-minute intervals. The gathered samples were centrifuged at 4000 rpm to remove suspended

nanoparticles before analysis. The residual content of MB (methylene blue) was determined by measuring absorbance with a UV–Vis spectrophotometer. The degradation efficiency (D%) of MB under UV irradiation

facilitated by green-synthesized CuO nanoparticles was assessed using the initial absorbance ( $A_0$ ) and the time-dependent absorbance ( $A_t$ ) at a specified irradiation duration ( $t$ ):

$$D\% = \frac{A_0 - A_t}{A_0} \% \quad \dots (1)$$

## 2.5 Preparation of CuO Nanoparticles-chitosan Nanocomposite

Two grams of produced CuO nanoparticles were uniformly dispersed in 100 mL of a chitosan solution, which was made with 1 wt% chitosan dissolved in 1 wt% acetic acid ( $\text{CH}_3\text{COOH}$ ). Mixture were incessantly agitated at 60 °C until a uniform suspension was attained, then underwent ultrasonication for 30 minutes to improve dispersion stability. The solution was then permitted to remain undisturbed at room temperature overnight to remove trapped air bubbles before further use.

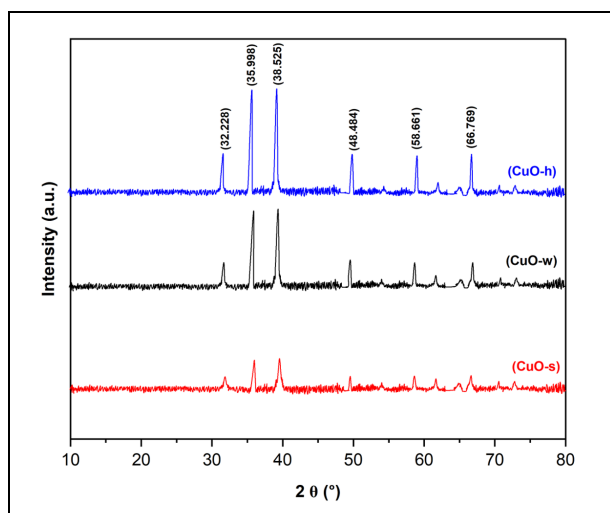


**Fig. 3: XRD patterns of CuO-s, CuO-w, and CuO-h nanoparticles calcined at 600 °C for 2 h, showing the characteristic diffraction peaks of monoclinic CuO**

Bleached and mercerized bamboo textiles served as the substrate for coating have been previously washed and dried at hotair oven at 40 °C for 8 minutes. A CuO loading of 2 wt.% relative to the chitosan solution was selected based on preliminary dispersion stability studies. Coating thickness was controlled through four consecutive dip-padding cycles under identical processing conditions. Fabric specimens (30 × 30 cm) were subsequently immersed four times at uniform chitosan solution containing well-dispersed CuO nanoparticles, whereas a control sample underwent same treatment with chitosan solution devoid of nanoparticles. The impregnated fabrics were then processed through a padded mangle at 35 rpm for 5 minutes to achieve uniform coating dispersion, followed by drying at 80 °C for 20 minutes. Untreated bamboo fabric (R-BF), chitosan-coated bamboo fabric (C-BF), and CuO

nanocomposite-coated bamboo fabric (CuO-C-BF) were prepared for comparative evaluation.

## 2.6 Water Repellent Property

The hydrophobicity of chitosan-treated bamboo textiles (C-BF) and CuO nanocomposite-coated (CuO-C-BF) was assessed by water contact angle with a VCA Optima system. A 5 µL water droplet was meticulously placed on five separate spots of each fabric sample to guarantee measurement accuracy. The contact angle was measured after 20 seconds of droplet placement, as untreated or minimally modified bamboo fabrics display only temporary resistance to wetting due to surface functional groups followed by rapid liquid penetration, which hinders precise WCA assessment and signifies intrinsic hydrophilic properties.

## 2.7 Antimicrobial Studies

### 2.7.1 Microbial Isolation and Culture Preparation

The cultures were reactivated and subcultured at 37 °C for 1 day, after which a loopful of each strain was inoculated into nutrient broth to generate fresh inoculum. Bacterial suspensions were incubated at 37 °C for 5–6 hours until turbidity indicated active growth, and then uniformly dispersed over solidified Mueller–Hinton agar plates using sterile swabs for additional antimicrobial analysis.

### 2.7.2 Agar Well Diffusion Technique

The efficacy of produced CuO NPs (CuO-s, CuO-w, and CuO-h) was qualitatively assessed against *Staphylococcus aureus* and *Escherichia coli* employing agar well diffusion method. Sterile distilled water served as the negative control, while a standard antibiotic disc served as the positive control. A volume of 100 µL of nanoparticle suspension was introduced into each well. Sterile Mueller–Hinton plates infected with bacterial cultures were perforated to make uniform wells of 7 mm diameter, into which varying quantities of nanoparticle suspensions (25, 50, and 100 mg mL<sup>-1</sup>) were inserted. The concentration range (25–100 mg mL<sup>-1</sup>) was selected based on preliminary antibacterial screening reports demonstrating concentration-dependent inhibition by CuO nanoparticles. The plates were subsequently incubated at 37 °C for 1 day, after which zones of inhibition in each well were measured in millimeters to assess antibacterial efficacy and to comparatively analyze the impact of synthesis methods on nanoparticle performance. Antibacterial efficacy of R-BF, C-BF, and CuO-C-BF was assessed against *Staphylococcus aureus* and *Escherichia coli* (Archana *et al.* 2021a). Fabric samples were excised into circular discs with a diameter of 10 mm and positioned onto freshly prepared Mueller–Hinton agar plates that were uniformly inoculated with corresponding bacterial cultures using sterile swabs.

Following a 15-minute drying period for the inoculation plates, the fabric discs were delicately pressed onto the agar surface under aseptic circumstances to ensure adequate contact. Each plate had three test samples and was incubated at 37 °C for 1 day, following which antibacterial efficacy was assessed by measuring the diameter of inhibitory zones surrounding the fabric discs.

### 2.7.3 Percentage Reduction Test

In accordance with the AATCC 100 standard, R-BF, C-BF, and CuO-C-BF were sectioned into circular specimens with a diameter of 10 mm and subjected to bacterial suspensions of *Escherichia coli* and *Staphylococcus aureus*. Following incubation at 37 °C for 24 h, surviving bacteria were serially diluted and plated on nutrient agar. Colony-forming units (CFU) were counted after incubation, and bacterial reduction percentages were calculated according to the AATCC 100 standard. The antibacterial efficacy was measured by the viable cell count technique by determining the percentage reduction in bacterial population according to standard evaluation protocols:

$$\text{Bacterial Reduction \%} = \left[ \frac{A-B}{A} \right] \times 100\% \quad \dots (2)$$

A signifies the initial viable bacterial cell count before exposure, while B indicates the remaining viable cell count following treatment with the fabric samples.

## 3. RESULTS AND DISCUSSION

CuO nanoparticles generated by a green method utilizing phytochemicals from *Hibiscus rosa-sinensis* leaf extract generally display low crystallinity or semi-amorphous traits in their initial condition, necessitating thermal treatment exceeding around 300 °C to achieve phase crystallization. Consequently, in this study, the produced nanoparticles were subjected to calcination at 600 °C to guarantee full crystallization and phase stability. The crystalline structure of the calcined CuO nanoparticles (CuO-s, CuO-w, and CuO-h) was validated by X-ray diffraction analysis, as illustrated in Fig. 3.

The diffraction peaks at 32.22°, 35.99°, 38.52°, 48.48°, 58.66°, and 66.76° correspond to the characteristic monoclinic CuO phase, confirming successful nanoparticle formation. The higher peak intensity observed for CuO-h indicates improved crystallinity compared with CuO-w and CuO-s. Crystallite size analysis revealed average values of 54.2, 40.3, and 30.1 nm for CuO-h, CuO-w, and CuO-s, respectively, demonstrating the influence of synthesis route on crystal growth.

The significant widening of Bragg reflections in the XRD patterns signifies the emergence of nanoscale CuO crystallites despite calcination. Average crystallite sizes were calculated using the Debye-Scherrer equation, expressed as equ. 2:

**Table 1. XRD and properties of CuO – S, CuO – W, and CuO H**

| (hkl)                    | FWHM (Radiation) |       |       | 2θ (°) | Crystallite Size, D (Nano Meter) |            |            |
|--------------------------|------------------|-------|-------|--------|----------------------------------|------------|------------|
|                          | CuO-S            | CuO-W | CuO-H |        | CuO-S (nm)                       | CuO-W (nm) | CuO-H (nm) |
| (110)                    | 0.280            | 0.210 | 0.155 | 32.228 | 29.8                             | 39.7       | 53.8       |
| (002)                    | 0.255            | 0.185 | 0.140 | 35.998 | 33.0                             | 45.5       | 60.1       |
| (111)                    | 0.240            | 0.175 | 0.132 | 38.525 | 35.4                             | 48.5       | 64.4       |
| (-202)                   | 0.310            | 0.235 | 0.170 | 48.484 | 28.3                             | 37.3       | 51.5       |
| (202)                    | 0.335            | 0.255 | 0.190 | 58.661 | 27.5                             | 36.1       | 48.5       |
| (-113)                   | 0.360            | 0.275 | 0.205 | 66.769 | 26.7                             | 35.0       | 46.9       |
| Average Crystallite Size |                  |       |       |        | 30.1                             | 40.3       | 54.2       |

$$D = \frac{K\lambda}{\beta \cos \theta} \quad \dots (3)$$

Where  $\lambda$  is the X-ray wavelength (0.1548 nm), K is the form factor (0.94),  $\beta$  signifies whole width at half maximum, and  $\theta$  indicates the diffraction angle. The determined crystallite sizes differed according to the

synthesis method, demonstrating that sonication-derived CuO (CuO-s) displayed the smallest average crystallite size (approximately 30.1 nm), followed by wet-chemical CuO (CuO-w, approximately 40.3 nm), whereas hydrothermally synthesized CuO (CuO-h) exhibited the largest crystallite size (approximately 54.2 nm), thereby

indicating that processing conditions substantially affect crystal growth and particle refinement.

The FTIR spectra of the calcined CuO nanoparticles (CuO-s, CuO-w, and CuO-h), subjected to heat treatment at 600 °C as depicted in Fig. 4, showcasing the distinctive vibrational bands linked to the CuO structure and remaining surface functional groups.

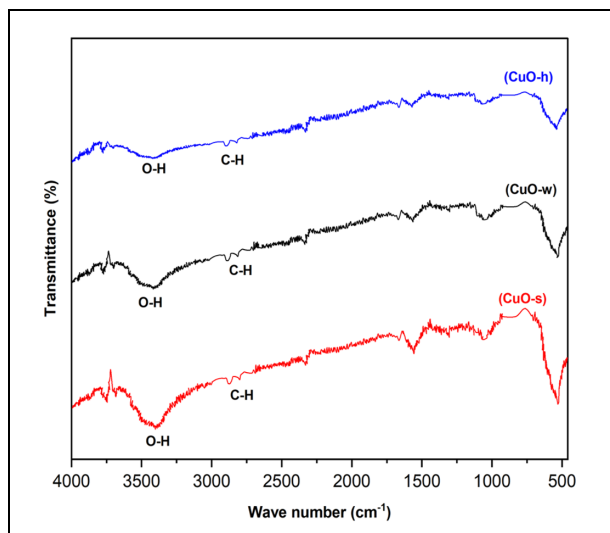


**Fig. 4:** FTIR spectra of CuO-s, CuO-w, and CuO-h nanoparticles calcined at 600 °C, illustrating characteristic Cu–O vibrations and surface functional groups

The FTIR spectra of CuO-s, CuO-w, and CuO-h exhibited similar vibrational features, including Cu–O stretching bands at 520–590  $\text{cm}^{-1}$  and broad O–H stretching bands associated with surface hydroxyl groups and adsorbed moisture. Additional bands observed in the regions of 2800–2950  $\text{cm}^{-1}$  and 1450–1550  $\text{cm}^{-1}$  are attributed to residual phytochemical species originating from the *Hibiscus rosa-sinensis* extract used during nanoparticle synthesis. The relatively stronger organic-related bands observed for CuO-s suggest a higher degree of surface-bound phytochemical residues rather than structural impurities. These results indicate that the synthesis route influences surface chemistry while preserving the characteristic CuO structure.

Elemental analysis via EDX verifies the presence of Cu and O as the principal components, indicating high compositional purity; however, the lack of detectable impurity elements contrasts with FTIR findings, suggesting that any residual organic species, if present are likely below EDX's detection threshold or exist solely on the surface.

The EDX spectra predominantly revealed the presence of Cu and O elements, indicating the successful formation of CuO nanoparticles. However, EDX has limited sensitivity toward light elements and trace surface species; therefore, the absence of additional

signals should not be interpreted as evidence of complete compositional purity.

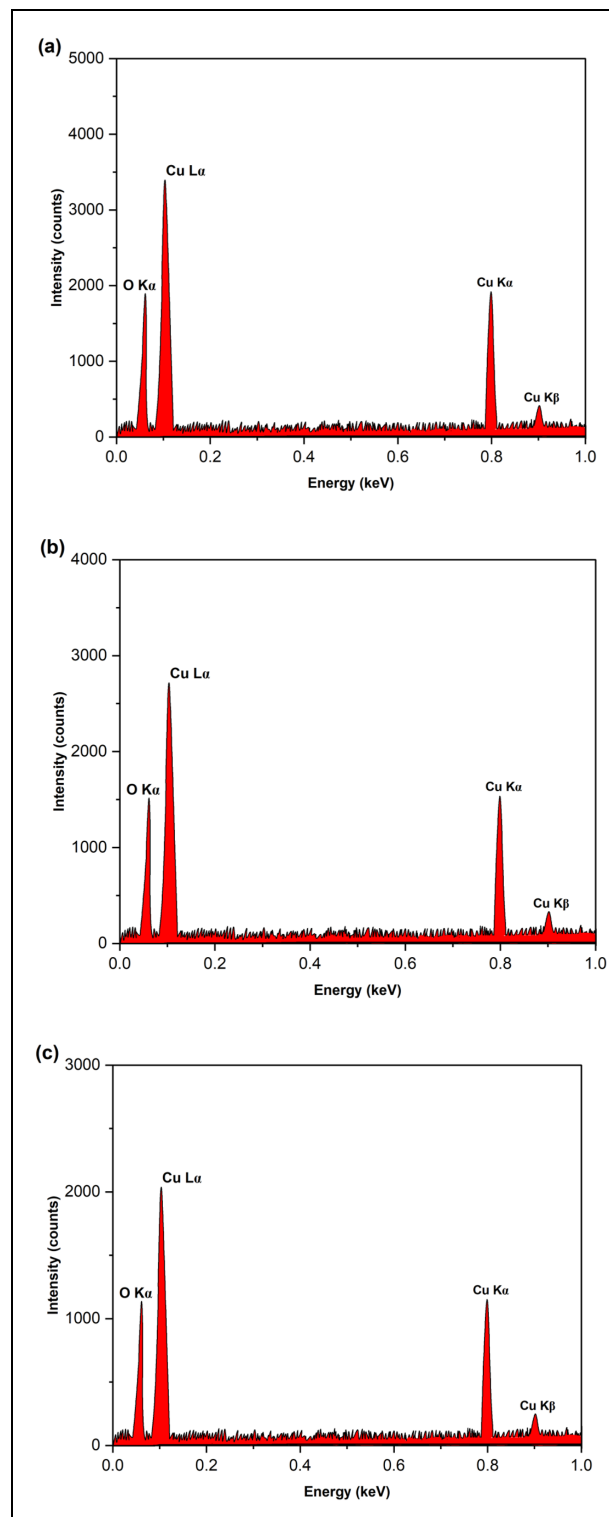


**Fig. 5:** EDX spectra of CuO nanoparticles (a) CuO-s, (b) CuO-w and (c) CuO-h

UV–Vis spectra of CuO nanoparticles produced with *Hibiscus rosa-sinensis* leaf extract are depicted in

Fig. 6, demonstrating their optical absorption characteristics across the examined wavelength range.

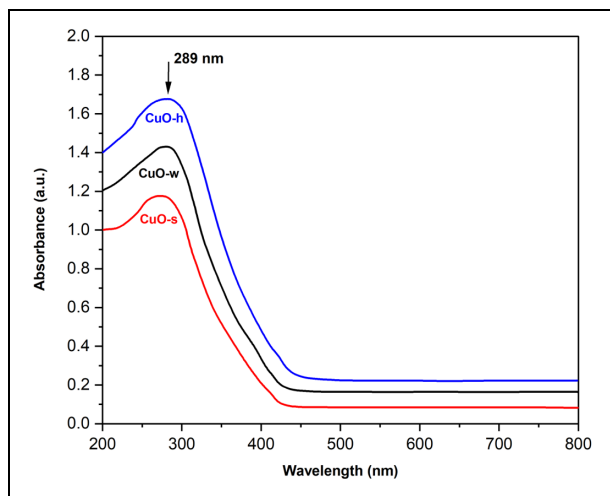


**Fig. 6: UV-Vis absorption spectra of CuO-s, CuO-w, and CuO-h nanoparticles recorded in the wavelength range of 200–800 nm**

The absorption band observed near 289 nm may be associated with charge-transfer transitions and surface-related electronic states influenced by nanoscale dimensions and phytochemical capping species. Similar blue-shifted absorption features have been reported for plant-mediated CuO nanostructures, where particle size and surface chemistry significantly affect optical behavior. The optical band gap was calculated utilizing Planck's equation:

$$E = hc/\lambda \quad \dots (4)$$

The optical band gap was further determined using the Tauc relation, by extrapolating the linear region of the absorption spectrum to the energy axis.

$$(ah\nu)^n = A(h\nu - E_g) \quad \dots (5)$$

where  $h$  shows Planck's constant,  $c$  denotes the speed of light, and  $\lambda$  signifies the absorption wavelength. The absorption edges were essentially constant across all samples, indicating similar optical absorption behavior among the synthesized CuO nanoparticles.

While the absorption maxima of CuO-s, CuO-w, and CuO-h nanoparticles remain relatively constant, a marked difference in absorption intensity is seen, with CuO-h demonstrating considerably more intensity than the other samples. This improvement is due to enhanced crystallinity, improved dispersion stability, and effective charge carrier dynamics, which facilitate stronger light-matter interaction. The enhanced optical response of CuO-h is associated with its improved crystal quality and reduced defect-related recombination, which facilitate more efficient charge separation and photocatalytic activity. These observations further demonstrate that

hydrothermally synthesized CuO nanoparticles exhibit a more refined size distribution and enhanced optical response compared to those produced through wet-chemical and sonication methods, supporting the trends identified in the XRD analysis and affirming the tunable optical properties of CuO in relation to synthesis parameters.

Subsequent to the XRD and EDX examinations, room-temperature photoluminescence (PL) investigations were conducted to assess defect states and potential ionic vacancies in the produced CuO nanoparticles (CuO-s, CuO-w, and CuO-h). A green emission band centered at approximately 470 nm was observed for all samples in Figure 7, indicating the presence of oxygen-vacancy-related defect states. Among the samples, CuO-h exhibited the lowest emission intensity, suggesting reduced defect density and improved structural quality.

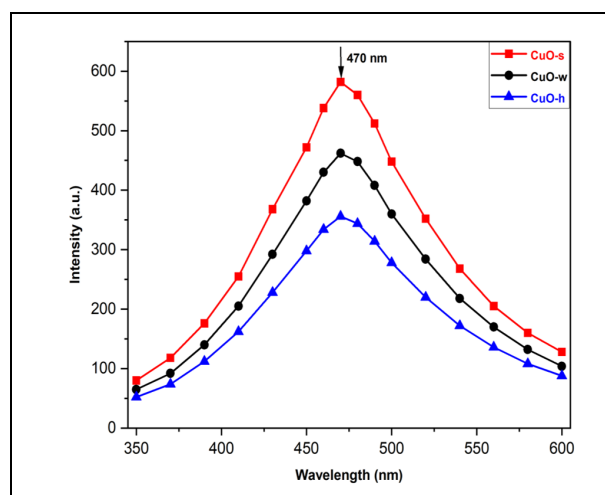


**Fig. 7: Room-temperature photoluminescence (PL) spectra of CuO-s, CuO-w, and CuO-h nanoparticles excited at 325 nm, recorded at room temperature (25 ± 2 °C)**

The PL results demonstrate that the excitonic emission location of the produced CuO nanoparticles is generally invariant to changes in synthesis procedure, processing conditions, surface area, or particle size, despite these factors having substantial impact on emission intensity. The lack of extensive emission bands in the photoluminescence spectra indicates few surface or bulk defect states, whereas the existence of a singular, sharp emission peak suggests that the nanoparticles primarily possess a restricted number of intrinsic structural defects, aligning with previously documented well-crystallized CuO nanostructures.

The green emission band observed around 470 nm is commonly associated with defect-related electronic transitions in CuO nanostructures, including possible oxygen-vacancy-related states reported in the literature. The lower PL intensity exhibited by CuO-h may indicate reduced defect-related recombination; however, direct

identification of specific defect species would require complementary analyses such as XPS or other defect-characterization techniques.

### 3.1 Photocatalytic Activity

Photocatalytic efficacy of green-synthesized CuO NPs (CuO-s, CuO-w, and CuO-h), produced using wet-chemical, hydrothermal, and sonication methods, was assessed by degradation of MB, serving as a model pollutant for wastewater treatment. Control studies showed that no substantial degradation transpired in the absence of UV irradiation or CuO nanoparticles, thus affirming that both light activation and the presence of a catalyst are crucial for efficient photocatalysis. Supplementary control tests demonstrated minimal photolysis of the dye only under UV light. The degradation was assessed by observing reduction at the distinctive absorption peak of MB at 665 nm, confirming that the noted decolorization is primarily facilitated by photocatalytic activity of CuO nanoparticles under UV light.

Figure 8 depicts the temporal evolution of UV–Vis spectra of aqueous MB in the presence of green-synthesized CuO nanoparticles (CuO-s, CuO-w, and CuO-h) under UV irradiation, demonstrating a gradual reduction in absorbance intensity with prolonged exposure time.

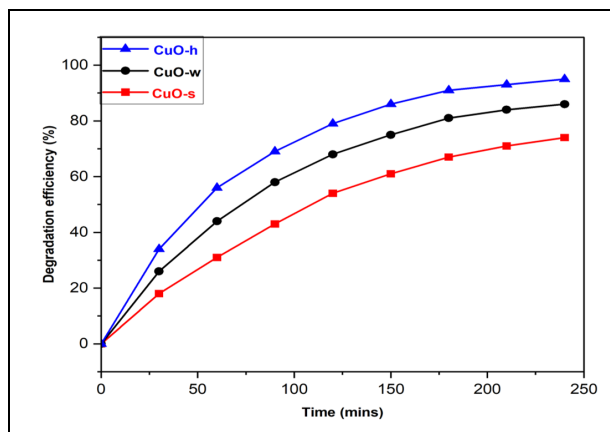


**Fig. 8: Photocatalytic degradation efficiency of methylene blue under UV irradiation using CuO-s, CuO-w, and CuO-h nanoparticles as photocatalysts**

The photocatalytic degradation of methylene blue is initiated by the absorption of UV light by CuO nanoparticles, resulting in the generation of electron-hole pairs ( $e^-/h^+$ ). The photogenerated electrons in the conduction band react with dissolved oxygen molecules to form superoxide radicals ( $\cdot O_2^-$ ), while the holes in the valence band interact with water molecules or hydroxyl

ions to produce hydroxyl radicals ( $\cdot OH$ ). These highly reactive oxygen species subsequently oxidize and decompose methylene blue molecules into smaller, less harmful intermediates and ultimately mineralize them into  $CO_2$  and  $H_2O$ . The superior photocatalytic activity of CuO – h may be attributed to its enhanced crystallinity and reduced defect-related recombination, which facilitate more efficient charge separation and prolong the lifetime of photogenerated charge carriers. The following reactions are:

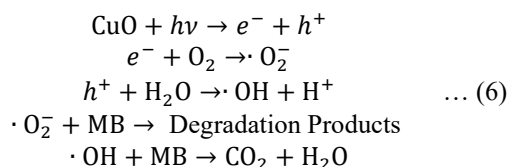


Figure 8 illustrates that the CuO nanoparticles (CuO-s, CuO-w, and CuO-h) display considerable photocatalytic efficacy in the breakdown of methylene blue under UV irradiation, as indicated by progressive increase in degradation efficiency. The degradation efficiency increased with irradiation time for all samples. CuO-h achieved the highest methylene blue degradation efficiency (~95%) after 240 min, followed by CuO-w (~86%) and CuO-s (~74%). The superior performance of CuO-h is attributed to its improved crystallinity, reduced defect density, and enhanced optical absorption.

Among the synthesized samples, the hydrothermally produced CuO-h nanoparticles demonstrate the highest photocatalytic efficiency with approximately 95% degradation of methylene blue within 240 minutes, in contrast to CuO-w (~86%) and CuO-s (~74%). The higher performance is due to enhanced crystallinity, improved optical response, and effective charge carrier separation of CuO-h, which promote increased surface reactivity and effective charge carrier separation. The results reveal that synthesis circumstances significantly affect catalytic behavior, and the synthesized CuO nanoparticles exhibit enhanced degrading efficiency compared to previously documented CuO-based systems and analogous nanomaterials.

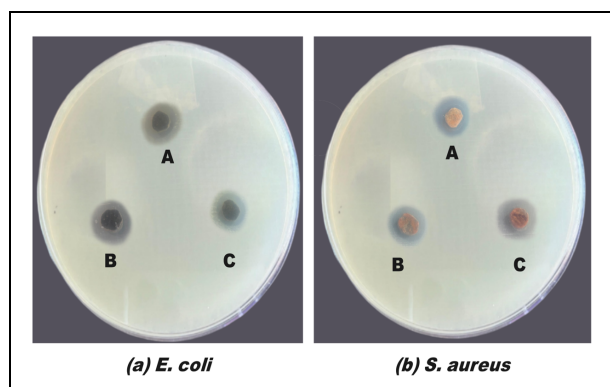
### 3.2 Antibacterial Activity of CuO Nanoparticles

The antibacterial assessment demonstrated that the inhibitory zones differ among the produced CuO nanoparticles (CuO-s, CuO-w, and CuO-h) when evaluated against *Escherichia coli* and *Staphylococcus aureus*. All samples showed antibacterial activity against Gram-negative and Gram-positive pathogens. Table 2 systematically presents the measured inhibitory zone sizes for various nanoparticle kinds and concentrations, emphasizing the comparative efficacy of each sample.

**Table 2. Antibacterial performance of CuO nanoparticles**

| Bacteria         | Concentration (mgL <sup>-1</sup> ) | CuO Nanoparticles |            |              |
|------------------|------------------------------------|-------------------|------------|--------------|
|                  |                                    | Wet-chemical      | Sonication | Hydrothermal |
| <i>E. coli</i>   | 25                                 | 8.2 ± 0.3         | 6.5 ± 0.2  | 11.4 ± 0.4   |
|                  | 50                                 | 13.6 ± 0.4        | 10.2 ± 0.3 | 16.8 ± 0.5   |
|                  | 100                                | 18.4 ± 0.5        | 15.1 ± 0.4 | 21.0 ± 0.6   |
| <i>S. aureus</i> | 25                                 | 9.5 ± 0.3         | 7.4 ± 0.2  | 12.8 ± 0.4   |
|                  | 50                                 | 15.8 ± 0.4        | 12.3 ± 0.3 | 18.9 ± 0.5   |
|                  | 100                                | 20.2 ± 0.5        | 17.1 ± 0.4 | 21.1 ± 0.6   |

Figure 9 depicts the antibacterial effectiveness of the produced CuO nanoparticles (CuO-s, CuO-w, and CuO-h) against *Staphylococcus aureus* and *Escherichia coli* at the maximum tested concentration (100 mg mL<sup>-1</sup>), shown by the respective zones of inhibition.

**Fig. 9: Inhibition zone of (a) CuO-w (b) CuO-h, and (c) CuO-s**

The hydrothermally produced CuO-h nanoparticles demonstrated the most extensive inhibitory zones, measuring 21.0 mm for *Escherichia coli* and 21.1 mm for *Staphylococcus aureus*. Comparative research reveals that nanoparticles synthesized by the hydrothermal process have enhanced antibacterial activity compared to those produced by sonication and wet-chemical techniques, and also surpass numerous previously documented CuO systems. The increased efficacy is mainly due to their enhanced crystallinity, improved surface activity, uniform aggregated morphology, and reduced defect density, which promote stronger interactions with bacterial cell membranes.

The superior antibacterial activity of CuO-h nanoparticles is primarily attributed to their improved crystallinity, reduced defect density, enhanced structural integrity, and favorable surface characteristics, which promote stronger interactions with bacterial cell membranes and improve the generation of reactive

oxygen species responsible for microbial inactivation. An increase in nanoparticle concentration leads to heightened antibacterial activity, as a greater quantity of CuO particles interacts with and disrupts microbial cells, establishing a direct correlation between bactericidal efficacy and concentration.

This study reveals that critical factors including surface area, particle size, shape, and dosage substantially affect the antibacterial efficacy of CuO nanoparticles. A thorough assessment of physicochemical, photocatalytic, and biological properties among the produced samples revealed that the hydrothermally synthesized CuO-h nanoparticles were the most successful, leading to their selection for further coating applications.

### 3.3 Characterization of Coated and Uncoated Fabrics

Scanning Electron Microscopy (SEM) micrographs illustrating the surface morphology of bamboo fabric specimens at varying stages of surface treatment, acquired in secondary electron imaging mode using a ZEISS scanning electron microscope.

Figure 10(a) shows the SEM micrograph of untreated bamboo fabric (R-BF), revealing its natural fibrous architecture with rough, striated fiber surfaces, distinct inter-fiber voids, and the absence of any surface coating. Naturally adhered micro-debris confirms the unmodified lignocellulosic nature of the raw fibers. Figure 10(b) presents the chitosan-coated bamboo fabric (C-BF), where a continuous chitosan layer partially covers the fiber surface, reducing surface roughness and filling inter-fiber voids. Minor pores and surface irregularities indicate the formation of a semi-dense chitosan network and successful coating deposition. Figure 10(c) illustrates the CuO nanocomposite-coated bamboo fabric (CuO-C-BF), characterized by a rougher and more heterogeneous surface due to the uniform

distribution of CuO nanoparticles within the chitosan matrix. Localized nanoparticle agglomerations and increased surface irregularity confirm the successful incorporation of CuO nanoparticles, resulting in a stable nanocomposite coating. In previous research, hybrid SiO<sub>2</sub>-CuO nanocomposite coatings exhibited improved corrosion resistance and hydrophobic behavior due to CuO incorporation. SEM confirmed uniform morphology, suggesting applicability in durable antibacterial coatings and protective functional surfaces (Khazaal *et al.* 2025).

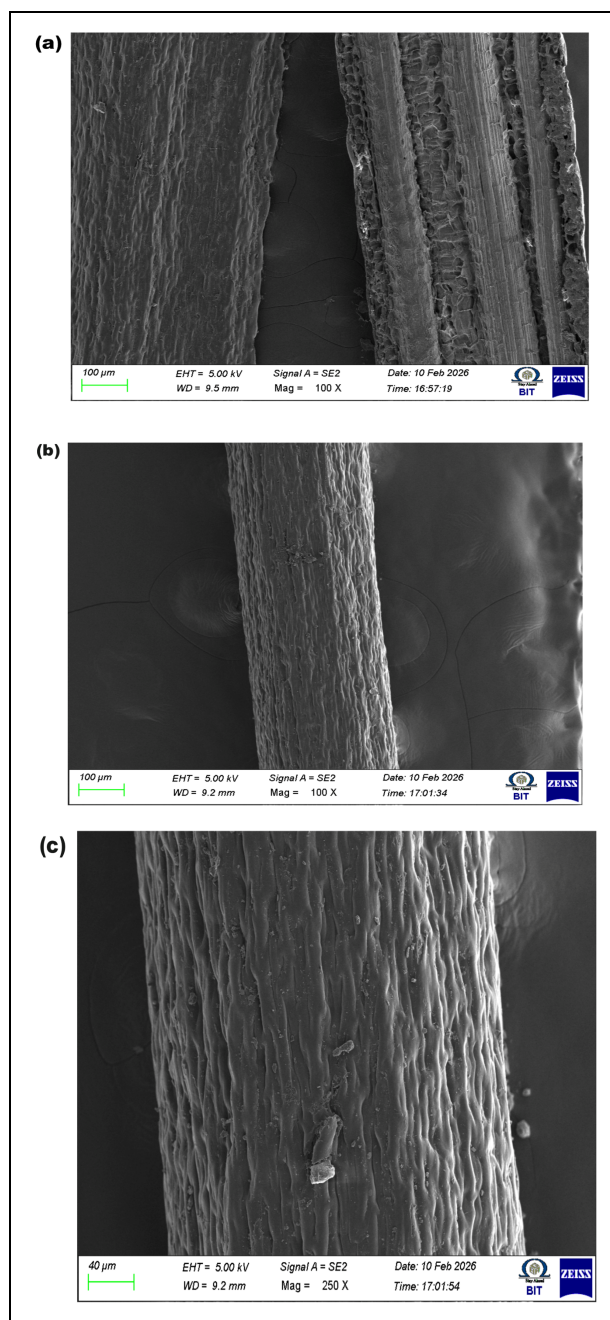


**Fig. 10: SEM Micrographs of (a) Untreated bamboo fabric (R-BF), (b) chitosan-coated bamboo fabric (C-BF) and (c) CuO nanocomposite-coated bamboo fabric (CuO-C-BF)**

### 3.4 Water Repellency of Fabric

A comparative evaluation of water repellency for untreated (R-BF), chitosan-coated (C-BF), and CuO nanocomposite-coated (CuO-C-BF) bamboo fabrics was performed utilizing the sessile drop method to measure the water contact angle. The uncoated cloth demonstrated quick water droplet absorption upon contact, demonstrating pronounced hydrophilic behavior, attributable to the prevalence of surface hydroxyl groups that facilitate rapid wetting and liquid infiltration.

In comparison to untreated fabric (R-BF), both chitosan-coated (C-BF) and CuO nanocomposite-coated (CuO-C-BF) samples demonstrate improved hydrophobic properties due to diminished surface energy and the establishment of a dense coating layer that partially occludes surface pores, as corroborated by SEM measurements. The uncoated cloth exhibits a contact angle of 0°, signifying total wettability, while C-BF displays moderate hydrophobicity ( $122 \pm 0.3^\circ$ ). Conversely, CuO-C-BF exhibits a markedly elevated contact angle of  $157 \pm 0.1^\circ$ , affirming its superhydrophobic properties. This enhancement is ascribed to the presence of finely dispersed CuO nanoparticles characterized by a large surface area and advantageous spherical aggregate form, which augment surface roughness and diminish liquid adhesion. Thus, the CuO/chitosan-coated bamboo fabric has enhanced water-repellent and self-cleaning capabilities relative to previously documented systems. According to previous studies, Zn-doped CuO nanostructures synthesized by the sol-gel method showed tunable particle size and improved optical properties, confirmed through XRD and SEM. Water contact angle analysis revealed a transition from hydrophobic to hydrophilic behavior, highlighting surface engineering potential for antibacterial coatings (Norelhouda *et al.* 2026).

### 3.5 Antibacterial Behavior of CuO Nanocomposite

Qualitative antibacterial efficacy of untreated (R-BF), chitosan-coated (C-BF), and CuO nanocomposite-coated (CuO-C-BF) fabrics was assessed by measuring the diameter of inhibition zones. The CuO-C-BF samples demonstrated the most extensive inhibitory zones against *Staphylococcus aureus* and *Escherichia coli*, signifying enhanced antibacterial activity, with the relevant values detailed in Table 3.

**Table 3. WCA for R- BF uncoated and CuO-C- BF and C- BF coated bamboo fabrics**

| Material  | Contact Angle |
|-----------|---------------|
| R- BF     | 0             |
| C- BF     | $122 \pm 0.3$ |
| CuO-C- BF | $157 \pm 0.1$ |

Table 4 and Fig. 11 illustrate the antibacterial efficacy of untreated (R-BF), chitosan-coated (C-BF), and CuO nanocomposite-coated (CuO-C-BF) fabrics as determined by inhibitory zone measurements. The CuO-C-BF samples display markedly bigger inhibition zones

than R-BF and C-BF, indicating superior antibacterial activity, with measurements of  $30.1 \pm 1.1$  mm against *Staphylococcus aureus* and  $21.0 \pm 1.1$  mm against *Escherichia coli*.

**Table 4. Antimicrobial performance of bamboo fabrics**

| Bacteria         | CuO Nanoparticles |                   |                   |
|------------------|-------------------|-------------------|-------------------|
|                  | R- BF             | C- BF             | CuO-C – BF        |
| <i>E. coli</i>   | -                 | $14.8 \pm 0.4$ mm | $21.0 \pm 1.1$ mm |
| <i>S. aureus</i> | -                 | $18.2 \pm 0.5$ mm | $30.1 \pm 1.1$ mm |

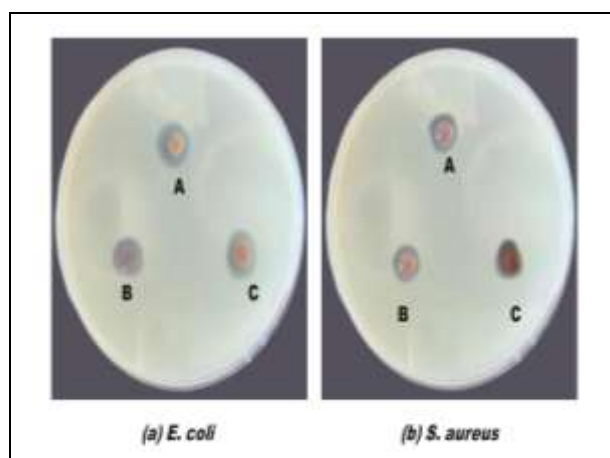


**Fig. 11: Inhibition Zone (a)R-BF (b) C-BF and (c) CuO-C-BF.**

A significant disparity in inhibition zone dimensions is noted, with CuO-C-BF demonstrating superior antibacterial efficacy against *Staphylococcus aureus* ( $30.1 \pm 1.1$  mm) in comparison to *Escherichia coli* ( $21.0 \pm 1.1$  mm). Untreated fabric (R-BF) exhibits no inhibitory zone, hence demonstrating the lack of intrinsic antibacterial activity in bamboo. The superior performance of CuO/chitosan-coated fabric is due to the efficient green synthesis of *Hibiscus rosa-sinensis* extract, resulting in highly crystalline CuO nanoparticles with improved surface activity and enhanced interaction with bacterial cells. In prior studies, green synthesis of metal oxide nanoparticles was achieved. The materials exhibited antimicrobial activity against *Escherichia coli* and *Staphylococcus aureus*, indicating potential for antibacterial and biomedical textile applications (Razack *et al.* 2020).

### 3.6 Percentage Reduction Test

A quantitative antibacterial assessment was performed utilizing the percentage reduction method to evaluate bacterial inhibition by untreated (R-BF), chitosan-coated (C-BF), and CuO nanocomposite-coated (CuO-C-BF) fabrics. Table 5 summarizes the comparative bacterial reduction efficacy against

*Escherichia coli* and *Staphylococcus aureus*, elucidating the usefulness of coatings.

**Table 5. Bacterial reduction of bamboo fabrics**

| Bacteria         | Bacterial Removal Efficiency (%) |                |                |
|------------------|----------------------------------|----------------|----------------|
|                  | R- BF                            | C- BF          | CuO-C – BF     |
| <i>E. coli</i>   | $8 \pm 0.5$ %                    | $87 \pm 0.8$ % | $96 \pm 0.6$ % |
| <i>S. aureus</i> | $11 \pm 0.4$ %                   | $90 \pm 0.7$ % | $99 \pm 0.3$ % |

The quantitative findings demonstrate that CuO nanocomposite-coated fabric (CuO-C-BF) attains exceptional bacterial reduction, achieving approximately  $96 \pm 0.6$  % efficacy against *Escherichia coli* and  $99 \pm 0.3$  % against *Staphylococcus aureus* in contrast to chitosan-coated fabric (C-BF), which exhibits reductions of  $87 \pm 0.8$  % and  $90 \pm 0.7$  %, respectively, while the untreated fabric shows minimal antibacterial activity. Similarly, the photocatalytic degradation efficiency ( $\sim 95\%$ ) achieved by the CuO nanoparticles demonstrates competitive performance relative to previously reported metal oxide-coated textile systems. These findings indicate that the developed CuO–chitosan-coated bamboo fabric possesses significant potential for antimicrobial, self-cleaning, and protective textile applications. The improved performance is due to the uniform dispersion of CuO nanoparticles on the fabric surface, which promotes effective interaction with leading to membrane disruption, bacterial cells, oxidative stress, and inhibition of cellular processes like DNA replication, aided by the advantageous structural morphology of the synthesized nanoparticles.

Overall, the synthesis route significantly influenced the physicochemical and functional properties of the CuO nanoparticles. The hydrothermal method produced CuO-h nanoparticles with improved

crystallinity, reduced defect-related recombination, and enhanced optical absorption, which collectively contributed to superior photocatalytic degradation and antibacterial performance. In contrast, the wet-chemical and sonication methods resulted in comparatively lower structural quality and functional efficiency. These findings demonstrate a strong structure–property–performance relationship, highlighting the critical role of synthesis conditions in determining the multifunctional behavior of green-synthesized CuO nanoparticles.

#### 4. CONCLUSION

In this study, CuO nanoparticles were successfully synthesized using *Hibiscus rosa-sinensis* leaf extract through wet-chemical, hydrothermal, and sonication-assisted routes. The results demonstrated that the synthesis method significantly influenced the structural, optical, photocatalytic, and antibacterial properties of the nanoparticles. Among the investigated methods, the hydrothermal route produced CuO nanoparticles with higher crystallinity, reduced defect-related recombination, and improved optical absorption characteristics. Consequently, the hydrothermally synthesized nanoparticles exhibited superior photocatalytic degradation of methylene blue and stronger antibacterial activity against both *Escherichia coli* and *Staphylococcus aureus* compared with the other synthesis routes. The optimized CuO nanoparticles were successfully incorporated into a chitosan matrix and applied onto bamboo fabric. The resulting coated fabric exhibited strong antibacterial activity, high photocatalytic degradation efficiency, and a high water contact angle ( $157^\circ$ ), indicating effective multifunctional behavior. These findings demonstrate the potential of green-synthesized CuO nanoparticles for the development of sustainable antimicrobial and water-repellent textile materials. However, the present study was limited to the evaluation of structural, optical, antibacterial, photocatalytic, and water-repellent properties. The durability of the coating, wash resistance, mechanical stability, long-term antibacterial performance, and advanced surface characterization were not investigated. Future studies should focus on these aspects to further assess the practical applicability and long-term performance of CuO-functionalized bamboo textiles for biomedical and protective textile applications.

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#### CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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