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Low-Temperature Chemical Precipitation Synthesis of Pure Monoclinic CuO (Copper Oxide) Nanoparticles

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Abstract: A low-temperature chemical precipitation (alkaline precipitation) route suitable for scalable production synthesized copper oxide (CuO) nanoparticles. Aqueous copper nitrate was reacted with sodium hydroxide under strongly alkaline conditions (pH ≈ 14) to form a $Cu(OH)_2$ precursor, followed by washing, drying (120 °C) and thermal dehydration (200 °C) to obtain CuO nanoparticles. Phase identification and crystallinity were examined by X-ray diffraction (XRD), functional group/bond confirmation by Fourier transform infrared spectroscopy (FTIR), and morphology by field-emission scanning electron microscopy (FESEM). XRD patterns confirmed the formation of phase-pure monoclinic tenorite CuO with no detectable impurity phases. The average crystallite size was estimated as ~49.01 nm using the Debye–Scherrer equation. FTIR spectra showed characteristic Cu–O stretching vibrations (≈500–600 cm^{-1}), supporting complete oxide formation and phase purity. FESEM images indicated nearly spherical CuO nanoparticles with relatively uniform distribution and minor agglomeration, consistent with rapid nucleation under strong alkalinity. Overall, the results demonstrate that alkaline chemical precipitation followed by mild heat treatment is a simple, low-cost and reproducible method for producing phase-pure CuO nanoparticles, providing a reliable materials platform for catalysis, sensing and energy-related applications.

Keywords: Copper Oxide (Cuo) Nanoparticles, Chemical Precipitation, Alkaline Precipitation, Monoclinic Tenorite Cuo, XRD–FTIR–FESEM Characterization, Debye–Scherrer Crystallite Size.

1. Introduction

Since materials at the nanoscale have properties that differ greatly from those of their bulk counterparts, nanomaterials have drawn a lot of attention in recent decades. Quantum confinement effects, a high surface-to-volume ratio, and changed defect chemistry take over the behavior of particles when their sizes are lowered below 100 nm [1, 2]. Nanostructured materials are very appealing for next-generation technologies because of these modifications, which affect optical absorption, electrical conductivity, catalytic activity, and magnetic response. The development of controlled synthesis techniques, especially bottom-up chemical methods, has made it possible to create nanomaterials with customized morphology and crystallinity under comparatively mild processing conditions [2]. Metal oxide nanoparticles such as Fe_2O_3, ZnO, TiO_2, CeO_2, and CuO have been widely explored for applications in catalysis, sensing, energy conversion, and environmental remediation because of their structural stability and tunable electronic structures [3, 4]. Copper oxide (CuO) has become one of the most significant transition metal oxides. CuO crystallizes in a monoclinic structure and is a p-type semiconductor with a narrow band gap of roughly 1.2 eV. Strong visible light absorption is made possible by its narrow band gap, which is advantageous for photovoltaic and photocatalytic applications. CuO also has good chemical stability and favourable redox characteristics, which support its application in gas sensors, supercapacitors, lithium-ion batteries, and antimicrobial systems [5, 6]. Compared to bulk CuO, the electrochemical and catalytic performance of nanocrystalline CuO is greatly improved by its increased surface area and altered electronic structure [7]. The controlled synthesis of CuO nanostructures with specific particle size, morphology, and phase purity has been the subject of much research due to these benefits.

Sol-gel processing, hydrothermal and solvothermal techniques, thermal decomposition, microwave-assisted synthesis, green synthesis using plant extracts, and chemical precipitation are some of the synthesis routes that have been documented for the production of CuO nanoparticles. Highly crystalline nanostructures can be produced using sol-gel and hydrothermal processes, but these methods frequently call for high temperatures, lengthy reaction times, and specialized autoclaves, raising operating costs [8]. Although they require regulated instrumentation and microwave parameter optimization, microwave-assisted routes offer uniform nucleation and quick heating [9]. Because biological extracts are environmentally friendly, green synthesis techniques have drawn attention; however, reproducibility and particle size control are still difficult in many situations [3, 10]. On the other hand, one of the simplest and most scalable methods for creating CuO nanoparticles is the chemical precipitation method. Under controlled pH conditions, copper salts and alkaline precipitants typically react to form copper hydroxide intermediates, which are then dried or calcined to produce copper oxide (CuO). Low synthesis temperature, easy experimental setup, high yield, and suitability for large-scale production are some of the benefits of this approach [11]. Moreover, crystallite size, defect density, and surface morphology are all significantly influenced by process variables like calcination temperature, stirring time, pH, and precursor concentration [12]. Research has demonstrated that monoclinic CuO with high crystallinity and few secondary phases can be produced by alkaline precipitation under controlled conditions [7].

In order to verify phase formation and nanoscale characteristics of synthesized CuO, structural and spectroscopic characterization methods are crucial. Fourier transform infrared (FTIR) spectroscopy detects distinctive Cu–O vibrational modes that verify phase purity, while X-ray diffraction (XRD) analysis uses the Debye–Scherrer equation to provide information on crystallographic structure and average crystallite size [13, 14]. Field emission scanning electron microscopy (FESEM), which aids in comprehending nucleation behavior and agglomeration tendencies in metal oxide nanoparticles, is frequently used to analyze surface morphology and particle distribution [15]. Correlating synthesis conditions with structural quality requires such thorough characterization. The significance of controlled nanostructure engineering to improve electrochemical and energy storage properties has also been emphasized by recent studies. Charge transfer processes and capacitance behaviour in transition metal oxides have been demonstrated to be strongly influenced by tailoring crystallite size and defect chemistry [16]. Smaller crystallite sizes and uniform morphology in CuO systems typically result in increased active surface area and better electron transport pathways, both of which are essential for catalytic and sensing applications [5]. Thus, creating a cost-effective and repeatable synthesis method that reliably produces phase-pure CuO nanoparticles continues to be a key research goal.

CuO nanoparticle synthesis has been reported in many studies, but there is still a practical need for simplified techniques that combine low energy consumption, minimal chemical usage, and dependable phase control. There is little discussion of the basic nucleation and growth behaviour under strongly alkaline precipitation conditions, despite the fact that many reported methods place a strong emphasis on application performance. Understanding the formation of crystallites and the morphological evolution of CuO nanostructures can be improved by methodically examining a simple precipitation route and then applying a moderate amount of heat treatment. In the present study, CuO nanoparticles were created using a straightforward and reasonably priced precipitation method in a controlled alkaline environment. Complete phase transformation was then achieved by thermal treatment. The goal of the study is to show that such a simple method can produce monoclinic, phase-pure CuO nanoparticles with uniform morphology and nanocrystalline dimensions. The synthesis strategy has been validated through extensive structural, spectroscopic, and morphological analyses. By focusing on simplicity, reproducibility, and scalability, this investigation seeks to provide a reliable foundation for further application-oriented research on CuO nanomaterials in energy, sensing, and catalytic domains.

2. Experimental

2.1 Materials and Reagents

Copper Nitrate Hexahydrate ($Cu(NO_3)_3.3H_2O$, 98%) and Sodium Hydroxide Pellet (NaOH, 97%) were acquired from SRL, distilled water and Ethanol from Changshu Hongsheng Fine Chemical Co., Ltd. High analytical purity chemicals were acquired and used for the analysis without any additional processing.

2.2 Preparation of Copper Oxide Nanoparticles

Strongly alkaline conditions were used to create copper oxide nanoparticles using a regulated chemical precipitation process. Because it allows for quick nucleation, a low processing temperature, minimal equipment needs, and scalability for large-scale production, the precipitation method is widely acknowledged as a feasible bottom-up approach for creating transition metal oxide nanomaterials.

Precipitation reactions proceed under atmospheric pressure and do not require specialized reactors, which makes the process economically advantageous for industrial adaptation when compared to hydrothermal or solvothermal techniques [2, 7]. Additionally, by carefully adjusting the precursor concentration, pH, and post-treatment conditions, the method enables systematic control over crystallite size and morphology [11].

In the present synthesis, copper nitrate hex hydrate was selected as the copper precursor due to its high solubility in water and complete dissociation into Cu^{2+} ions, which ensures uniform distribution of metal ions in solution. A 0.1 M aqueous solution of copper nitrate was prepared using distilled water and stirred continuously at room temperature to obtain a homogeneous reaction medium. Sodium hydroxide (1.0 M) was then introduced dropwise under vigorous magnetic stirring until the solution reached a pH of approximately 14. The gradual addition of the alkaline solution is critical because it controls the supersaturation level and promotes uniform nucleation rather than uncontrolled particle growth. Under these conditions, Cu^{2+} ions react with OH^- ions to form copper hydroxide ($Cu(OH)_2$) as an intermediate precipitate according to the reaction:

$$Cu^{2+} + 2OH^- \rightarrow Cu(OH)_2 \downarrow$$

The formation of a bluish precipitate indicates the generation of copper hydroxide nuclei. Sustained stirring for two hours ensures complete precipitation and allows uniform growth of the nuclei throughout the reaction medium. In strongly alkaline environments, rapid nucleation dominates over crystal growth, leading to the formation of fine nanoscale particles. Such high-pH precipitation conditions have been reported to favour the production of smaller and more uniform CuO crystallites after thermal treatment [17]. After precipitation, centrifugation was used to separate the solid product, and ethanol and distilled water were used for repeated washings. In order to eliminate excess sodium ions and residual nitrate ions that might otherwise remain adsorbed on the particle surface, the washing step is essential. If ionic species are not completely removed, the nanoparticles' surface chemistry may be impacted or impurity phases may result. Thorough washing improves phase purity and crystallinity of the final CuO product after calcination, according to prior research [5].

The washed precipitate was dried at 120 °C for 4 hours to remove physically adsorbed water and to initiate partial dehydration of copper hydroxide. Subsequent thermal treatment at 200 °C for 2 hours facilitated the transformation of $Cu(OH)_2$ into CuO according to the reaction:

$$Cu(OH)_2 \rightarrow CuO + H_2O$$

This dehydration process results in the formation of monoclinic CuO nanoparticles (Figure 1). The selected calcination temperature was sufficient to achieve complete phase conversion while avoiding excessive grain growth or sintering, thereby preserving nanoscale features. Literature reports confirm that moderate annealing temperatures in the range of 200–400 °C are effective for obtaining phase-pure CuO from hydroxide precursors without inducing structural coarsening [18]. The simplicity of this precipitation route lies in its ambient pressure operation, aqueous reaction medium, short reaction duration, and minimal chemical requirements. The current method only uses controlled pH adjustment and thermal dehydration, in contrast to hydrothermal or microwave-assisted methods, which might need sealed autoclaves or specialized energy sources [19]. This kind of operational simplicity facilitates easy scale-up and improves reproducibility. Such operational simplicity enhances reproducibility and allows straightforward scale-up. Additionally, the strong alkaline environment ensures rapid nucleation, which contributes to uniform crystallite formation and controlled particle growth.

Compatibility with large-scale production is another benefit of the chosen strategy. The method is appropriate for industrial-level synthesis since precipitation reactions can be carried out in large volumes of solution without requiring major changes to process parameters. The scalability potential of precipitation-based CuO production is further demonstrated by rotating packed bed and continuous precipitation systems documented in previous works [20]. As a result, the approach used in this study is consistent with future practical applications as well as laboratory-scale research. Overall, controlled nucleation, efficient precursor conversion, and the formation of nanocrystalline CuO under mild thermal conditions are guaranteed by the simple precipitation method used here. Phase-pure monoclinic CuO nanoparticles with repeatable structural properties are produced by combining high pH precipitation, methodical washing, and moderate annealing. This synthesis strategy provides a reliable and economically viable pathway for producing CuO nanomaterials suitable for structural, catalytic, sensing, and electrochemical applications.

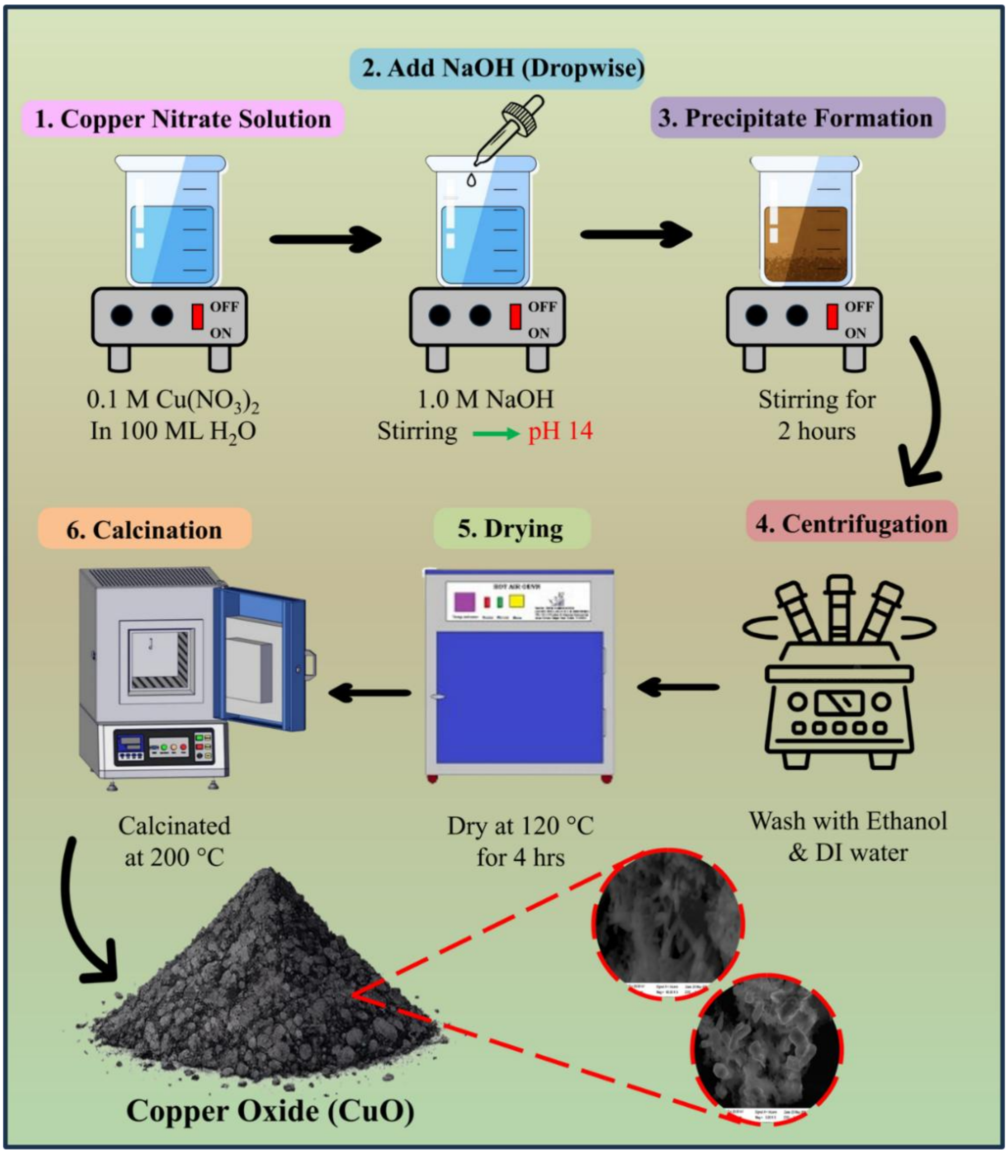

Figure 1. Reaction mechanism of CuO nanoparticles

2.3 Instrumentation

To identify phase purity and crystallinity, X-ray diffraction (XRD) was performed using a Malvern Panalytical Empyrean Series III diffractometer with a Cu Kα radiation source (λ = 1.54 Å). A PerkinElmer Spectrum Two model with a wavelength range of 4000 cm^{-1} to 400 cm^{-1} was used for FT-IR spectroscopy. A Carl Zeiss Sigma 300 (Germany) with a resolution of 1.0 nm at 15 kV and 1.6 nm at 1 kV and a magnification capability of up to 1,000,000 times was used to record the FESEM images.

3. Characterisation

The crystal structure of nanoparticles is investigated by X-ray diffraction, while their functional groups are identified by Fourier Transform Infrared Spectroscopy (FTIR). Surface morphological particle size and shape can be identified using Field Emission Scanning Electron Microscopy (FESEM).

3.1. XRD

The crystalline structure and phase purity of the synthesised copper oxide nanoparticles were systematically analysed using X-ray diffraction (XRD). XRD remains one of the most reliable and widely adopted techniques for determining crystallographic phase, lattice symmetry, crystallite size, and structural defects in nanomaterials. In transition metal oxides, subtle changes in peak position, intensity, and broadening often provide critical insights into phase evolution and nanoscale effects. The diffraction pattern obtained for the present CuO sample, recorded in the 2θ range of 10°–80° using Cu Kα radiation (λ = 1.5406 Å), clearly demonstrates the formation of a well-crystalline copper oxide phase.

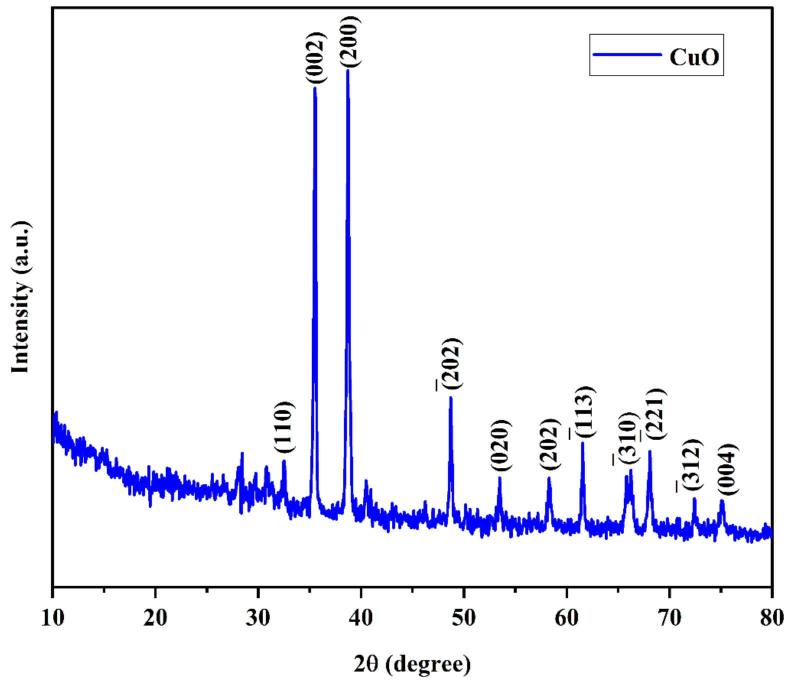

Figure 2 XRD pattern of Copper Oxide nanoparticl

The observed diffraction peaks (Figure 2) located at approximately 2θ = 32.5°, 35.5°, 38.7°, 48.7°, 53.5°, 58.3°, 61.5°, 66.2°, 68.0°, 72.4°, and 75.0° can be indexed to the (110), (–111)/(002), (111)/(200), (–202), (020), (202), (–113), (–311), (220), (–312), and (004) crystallographic planes of monoclinic CuO, respectively. These reflections are in good agreement with the standard data for tenorite CuO (JCPDS card no. 89-2531), confirming that the synthesised material crystallises in the monoclinic structure with space group C2/c. Similar diffraction signatures for monoclinic CuO nanoparticles synthesised through solution-based routes have been reported in earlier studies [7, 11]. The absence of additional diffraction peaks corresponding to Cu_2O or metallic Cu phases indicates that the precipitation process followed by thermal treatment has resulted in phase-pure CuO without secondary impurities. Such phase purity is particularly important because even trace Cu_2O phases can significantly influence the optical and electrochemical behaviour of copper oxide systems [5].

The sharpness and intensity of the dominant peaks, especially those corresponding to the (–111)/(002) and (111)/ (200) planes, indicate good crystallinity of the nanoparticles. At the same time, noticeable peak broadening is observed compared to bulk CuO, which is a characteristic feature of nanoscale materials. Peak broadening in XRD patterns arises mainly from finite crystallite size and lattice strain effects. The average crystallite size (D) was estimated using the Debye–Scherrer equation:

$$D = (K\lambda) / (\beta \cos \theta)$$

where K is the shape factor (commonly taken as 0.9–0.94), λ is the wavelength of the incident X-ray, β is the full width at half maximum (FWHM) of the selected diffraction peak (in radians), and θ is the Bragg diffraction angle. The Debye–Scherrer approach remains widely used for first-level crystallite size estimation in nanostructured oxides [13]. Using the most intense diffraction peak for calculation, the average crystallite size of the prepared CuO nanoparticles was found to be approximately 49 nm, confirming the nanocrystalline nature of the material.

It is crucial to remember that the crystallite size determined by XRD correlates to coherent diffraction domains and could be smaller than the particle size seen by electron microscopy because of potential agglomeration. Metal oxide nanoparticles frequently exhibit these variations, as high surface energy encourages particle clustering without necessarily changing the internal crystalline domains [15]. Uniform nucleation and controlled growth during the precipitation process are suggested by the comparatively narrow size distribution deduced from the steady peak broadening. Preferential orientation can also be inferred from the relative intensity ratios of the prominent peaks. Strong texturing effects were not present in this instance, as evidenced by the lack of aberrant enhancement or suppression of particular reflections. This suggests that the nanoparticles are randomly oriented, which is typical for powders synthesised via chemical precipitation methods [21]. The successful formation of a single-phase monoclinic CuO structure further confirms that the strongly alkaline environment (pH ≈ 14) facilitated complete conversion of copper nitrate precursor into copper hydroxide intermediate, followed by its dehydration into CuO during thermal treatment.

The structural integrity of monoclinic CuO is particularly relevant for applications in catalysis and electrochemical energy storage. The specific arrangement of Cu and O atoms in the monoclinic lattice governs charge transport behaviour and surface reactivity. Studies have shown that well-crystallised monoclinic CuO nanoparticles exhibit enhanced electrochemical performance due to improved electron mobility and stable redox transitions between Cu^{2+} and Cu^{1+} states [22]. Therefore, confirmation of phase-pure monoclinic CuO through XRD analysis is not merely a structural validation step but also a crucial indicator of potential functional performance. The XRD analysis clearly establishes that the adopted precipitation route, combined with controlled thermal annealing, leads to the formation of nanocrystalline, phase-pure monoclinic CuO. The absence of secondary phases, good crystallinity, and nanoscale crystallite size collectively demonstrate that the synthesis strategy is efficient and reproducible. These structural characteristics provide a strong foundation for subsequent morphological and spectroscopic analyses and support the suitability of the prepared CuO nanoparticles for advanced technological applications.

3.2 FTIR

Fourier Transform Infrared (FTIR) spectroscopy was employed to investigate the chemical bonding, functional groups, and phase formation of the synthesised copper oxide nanoparticles. FTIR analysis is particularly useful in metal oxide systems because it confirms the formation of characteristic metal–oxygen bonds and also detects surface-adsorbed species that may influence catalytic or electrochemical behaviour. For nanomaterials prepared via wet chemical routes, FTIR additionally provides indirect information about precursor conversion and the removal of residual organic or nitrate species after calcination. The FTIR spectrum (as shown in figure 3) recorded in the range of 4000–400 cm^{-1} shows distinct absorption bands corresponding to both Cu–O lattice vibrations and minor surface species. The most important features of the spectrum are the strong absorption bands observed at approximately 593 cm^{-1} and 532 cm^{-1}. These bands are attributed to the stretching vibrations of Cu–O bonds in monoclinic CuO. In copper oxide, the Cu–O vibrational modes typically appear in the 500–600 cm^{-1} region, and their presence is considered a direct confirmation of CuO phase formation [13]. Similar vibrational assignments for Cu–O stretching in nanocrystalline CuO synthesised through chemical routes have been consistently reported in earlier studies [15].

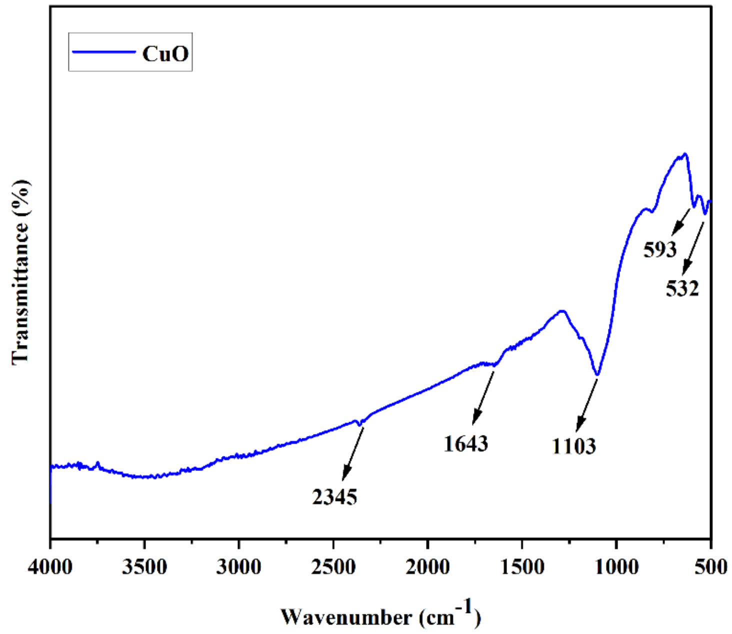

Figure 3. FTIR Spectrum of Copper Oxide nanoparticle

The appearance of two closely spaced bands in this region is consistent with the low-symmetry monoclinic structure of CuO, where different Cu–O bond environments lead to multiple infrared-active modes The intensity and sharpness of the Cu–O bands indicate good crystallinity and effective conversion of copper precursor into oxide form during thermal treatment. Incomplete conversion or the presence of copper hydroxide intermediates would typically result in additional bands at higher wavenumbers associated with O–H bending or nitrate vibrations.

The absence of strong nitrate-related peaks in the 1300–1500 cm^{-1} region suggests that the washing and annealing steps were sufficient to remove residual precursor species. This observation is in agreement with XRD results, which confirmed phase-pure monoclinic CuO without secondary phases [7]. A moderate absorption band observed around 1103 cm^{-1} can be attributed to C–O or C=O stretching vibrations of adsorbed carbonaceous species. Such features are commonly detected in nanoparticles synthesised through aqueous precipitation methods, as atmospheric CO_2 and trace organic impurities may adsorb onto high-energy nanoparticle surfaces during drying [3]. The presence of weak carbon-related bands does not indicate impurity phases within the crystal lattice but rather surface adsorption, which is typical for nanoscale metal oxides due to their high surface area.

The band appearing near 1643 cm^{-1} corresponds to the bending vibration of molecular water (H–O–H). This peak indicates the presence of physically adsorbed moisture on the nanoparticle surface. Nanocrystalline CuO, owing to its enhanced surface area, can readily adsorb atmospheric moisture, and such bending vibrations are commonly reported in chemically synthesised oxide nanoparticles [5]. The absence of a broad and intense O–H stretching band around 3400 cm^{-1} suggests that hydroxyl groups are present only in small amounts, confirming effective dehydration during calcination at elevated temperature. A weak absorption band observed near 2345 cm^{-1} is typically associated with asymmetric stretching vibrations of CO_2 molecules from the ambient environment. This feature is frequently observed in FTIR spectra recorded under normal atmospheric conditions and does not correspond to intrinsic lattice vibrations of CuO [15]. The low intensity of this band further supports that the synthesised nanoparticles are largely free from significant carbonate contamination. The overall FTIR profile therefore confirms three important aspects: first, the successful formation of Cu–O bonds characteristic of monoclinic CuO; second, the absence of residual nitrate or hydroxide precursor species; and third, the presence of minor surface-adsorbed water and carbonaceous molecules, which are common in nanomaterials prepared through precipitation routes. The vibrational features observed are fully consistent with previously reported FTIR analyses of chemically synthesised CuO nanoparticles [11].

From a functional perspective, the confirmation of strong Cu–O bonding is significant because the electronic and catalytic behaviour of CuO strongly depends on its lattice integrity and oxygen coordination environment. Studies have shown that well-defined Cu–O bonding and minimal surface contamination enhance charge transport and redox activity in CuO-based electrochemical systems [23]. Therefore, the FTIR results not only validate the structural formation of CuO but also indirectly indicate the suitability of the prepared nanoparticles for advanced applications in sensing, catalysis, and energy storage. In summary, the FTIR investigation provides clear spectroscopic evidence of phase-pure monoclinic CuO formation through the adopted precipitation method. The presence of characteristic Cu–O stretching bands in the 500–600 cm^{-1} region, along with the absence of undesirable precursor-related vibrations, confirms the effectiveness of the synthesis and thermal treatment processes. These findings complement the XRD results and collectively establish the successful preparation of nanocrystalline CuO with appropriate structural and chemical integrity.

3.3. FESEM

The surface morphology and microstructural features of the synthesised CuO nanoparticles were examined using field emission scanning electron microscopy (FESEM). Morphological evaluation is essential in nanomaterials research because particle shape, size distribution, and agglomeration behaviour strongly influence surface area, catalytic activity, and electrochemical performance. In chemically synthesised metal oxides, nucleation rate, growth kinetics, and post-synthesis thermal treatment collectively determine the final morphology. Therefore, FESEM analysis provides critical insight into the effectiveness of the adopted precipitation route. Figure 4(a) presents the FESEM image recorded at a magnification of 5.00 kX with a scale bar of 2 µm. The micrograph reveals that the synthesised CuO consists of densely packed particles forming agglomerated clusters.

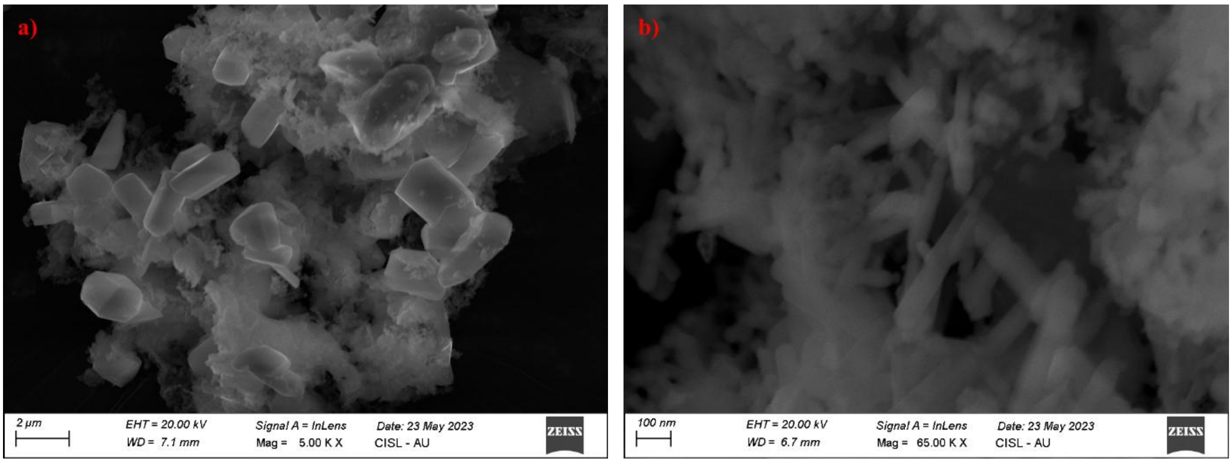

Figure 4. FESEM image of Copper Oxide at various magnifications

The agglomeration is not unexpected, as nanocrystalline metal oxides possess high surface energy and tend to minimise their total free energy through particle–particle interactions. Such clustering behaviour has been widely reported for chemically precipitated CuO nanoparticles and is mainly attributed to van der Waals attraction and surface hydroxyl interactions during drying [24]. Despite this agglomeration at the microscale, individual particles within the clusters appear well defined and exhibit relatively uniform morphology.

At higher magnification, as shown in Figure 4(b) (65.00 kX, 100 nm scale bar), the nanoparticles are more clearly resolved. The particles predominantly display short rod-like to slightly elongated or irregular polygonal morphologies. The formation of such anisotropic features can be linked to directional crystal growth along energetically favourable crystallographic planes of monoclinic CuO. In solution-based synthesis, rapid nucleation under strongly alkaline conditions followed by controlled growth often results in non-spherical morphologies, particularly when growth rates vary along different lattice directions [5]. The observed morphology therefore reflects the interplay between nucleation kinetics and subsequent crystallite growth during the precipitation and calcination steps.

The particle dimensions estimated from the high-magnification micrograph indicate that the primary crystallites are in the nanometre range, which is consistent with the average crystallite size of approximately 49 nm obtained from XRD analysis. It is important to distinguish between crystallite size derived from XRD and particle size observed in FESEM. While XRD provides the size of coherently diffracting domains, FESEM shows aggregated particle assemblies that may consist of one or multiple crystallites. Such correspondence between XRD and FESEM results confirms that the precipitation method successfully produced nanocrystalline CuO with limited grain coarsening during thermal treatment [13].

The surface texture observed in the micrographs appears relatively smooth with no visible secondary particulate phases. The absence of irregular large grains or phase-segregated domains further supports the phase purity indicated by XRD results. In chemically synthesised CuO systems, incomplete conversion or inadequate calcination may lead to residual $Cu(OH)_2$ or Cu_2O phases, which typically manifest as morphological heterogeneity [25]. The uniformity observed in the present FESEM images suggests that the adopted synthesis and annealing conditions were sufficient to achieve complete transformation into monoclinic CuO. The slight agglomeration seen at lower magnification can also be correlated with the high surface area and strong interparticle interactions typical of nanoscale oxides. Such agglomeration does not necessarily compromise functional performance; in many electrochemical and catalytic systems, loosely aggregated nanoparticles still provide accessible active sites due to porous cluster formation [26]. Moreover, precipitation-derived CuO nanoparticles are often reported to form interconnected networks, which can facilitate electron transport pathways in sensing and energy storage applications [27].

Another important observation from the FESEM analysis is the absence of excessive particle growth or sintering, even after calcination at elevated temperature. Excessive thermal treatment generally leads to grain enlargement and loss of nanoscale features. The retention of nanostructured morphology indicates that the chosen annealing temperature was adequate for phase conversion while preventing significant coalescence. This balance is crucial for preserving high surface-to-volume ratio, which directly influences catalytic and electrochemical efficiency in CuO-based systems [28]. The FESEM analysis demonstrates that the precipitation technique used in this work yields nanocrystalline CuO with regulated particle sizes and a comparatively uniform morphology. As is typical of chemically synthesized metal oxide nanoparticles, the particles have short rod-like to irregular nanoscale structures with moderate agglomeration. The efficacy of the synthesis strategy is validated by the morphological features' strong agreement with the crystallographic results obtained from XRD. The prepared CuO nanoparticles are attractive options for applications requiring high surface area, stable crystal structure, and effective charge transport because of these structural and surface features.

4. Conclusion

In this paper, copper oxide nanoparticles were synthesised by a simple precipitation method under alkaline conditions. The XRD study of the structure showed that the precipitation of monoclinic CuO with very high phase purity and an average crystallite size of around 49.01 nm, calculated from the Debye–Scherrer equation, indicates the successful creation of nanocrystalline material. The FTIR spectrum confirmed the formation of Cu–O bonds and indicated the presence of a few surface-adsorbed species, which in turn showed that the precursor materials had been formed effectively. FESEM images showed nanoparticles with nearly spherical shapes and slight, uniform agglomeration, which is typical of metal oxide nanoparticles due to strong surface interactions. In general, the results confirm that the precipitation route used was very efficient, reproducible, and capable of producing CuO nanoparticles with the required structural and morphological characteristics.

References

[1] R. Rajamani, C. Van Nguyen, Plasma-Assisted synthesis and engineering of functional nanomaterials: from metal nanoparticles to 2D architectures. NanoNEXT, 6(4), (2025) 10–47. https://doi.org/10.54392/nnxt2542

[2] A.S. John, K. Gurumurthy, Synthesis and Characterization of CuO Nanoparticles from Bioleached Copper through Modified and Optimized Double Precipitation Method. ACS Omega, 10(10), (2025) 10193–10198. https://doi.org/10.1021/acsomega.4c09064

[3] M.D. Moroda, T.L. Deressa, A.H. Tiwikrama, T.F. Chala, Green synthesis of copper oxide nanoparticles using Rosmarinus officinalis leaf extract and evaluation of its antimicrobial activity. Next Materials, 7, (2024) 100337. https://doi.org/10.1016/j.nxmate.2024.100337

[4] S. Srimathi, V. Kalaiselvi, P. Yasotha, B. Blessymol, S. Gopi, Plant-Mediated Synthesis of TiO_2–ZnO Nanocomposites Using Nigella sativa Seeds for Solar Energy Applications. NanoNEXT, 6(3), (2025) 1–8. https://doi.org/10.54392/nnxt2531

[5] T.H. Tran, V.T. Nguyen, Copper Oxide nanomaterials Prepared by solution methods, some properties, and potential applications: A Brief review. International Scholarly Research Notices, 2014, (2014) 1–14. https://doi.org/10.1155/2014/856592

[6] S. Rakshit, K.G. Mondal, B.S. Kar, T.N. Ghosh, D. Maji, M.N. Goswami, P.C. Jana, Enhanced structural, optical and electrical properties of Cu2O and CuO nanoparticles for electrochemical energy storage devices. Journal of Alloys and Compounds, 1024, (2025) 180274. https://doi.org/10.1016/j.jallcom.2025.180274

[7] C. Lin, J. Liao, Production of CuO nanoparticles using a simple precipitation method in a rotating packed bed with blade packings. Journal of Alloys and Compounds, 775, (2018) 419–426. https://doi.org/10.1016/j.jallcom.2018.09.187

[8] A. Vinukonda, N. Bolledla, R.K. Jadi, R. Chinthala, V.R. Devadasu, Synthesis of nanoparticles using advanced techniques. Next Nanotechnology, 8, (2025) 100169. https://doi.org/10.1016/j.nxnano.2025.100169

[9] Y. Li, Y. Lu, K. Wu, D. Zhang, M. Debliquy, C. Zhang, Microwave-assisted hydrothermal synthesis of copper oxide-based gas-sensitive nanostructures. Rare Metals, 40(6), (2020) 1477–1493. https://doi.org/10.1007/s12598-020-01557-4

[10] B. Cardoso, G. Nobrega, I.S. Afonso, J.E. Ribeiro, R.A. Lima, Sustainable green synthesis of metallic nanoparticle using plants and

microorganisms: A review of biosynthesis methods, mechanisms, toxicity, and applications. Journal of Environmental Chemical Engineering, 13(3), (2025) 116921. https://doi.org/10.1016/j.jece.2025.116921

[11] K. Phiwdang, S. Suphankij, W. Mekprasart, W. Pecharapa, Synthesis of CUO nanoparticles by precipitation method using different precursors. Energy Procedia, 34, (2013) 740–745. https://doi.org/10.1016/j.egypro.2013.06.808

[12] E.Y. Shaba, J.O. Jacob, J.O. Tijani, M.a.T. Suleiman, A critical review of synthesis parameters affecting the properties of zinc oxide nanoparticle and its application in wastewater treatment. Applied Water Science, 11(2), (2021). https://doi.org/10.1007/s13201-021-01370-z

[13] S. Suresh, S. Karthikeyan, K. Jayamoorthy, FTIR and multivariate analysis to study the effect of bulk and nano copper oxide on peanut plant leaves. Journal of Science Advanced Materials and Devices, 1(3), (2016) 343–350. https://doi.org/10.1016/j.jsamd.2016.08.004

[14] Y.S. Jara, E.T. Mohammed, T.T. Mekiso, Biosynthesized pure CuO, N-CuO, Zn-CuO, and N-Zn-CuO nanoparticles for photocatalytic activity: Enhanced optical properties through bandgap engineering. Next Materials, 8, (2025) 100742. https://doi.org/10.1016/j.nxmate.2025.100742

[15] M.S. Dehaj, M.Z. Mohiabadi, Experimental study of water-based CuO nanofluid flow in heat pipe solar collector. Journal of Thermal Analysis and Calorimetry, 137(6), (2019) 2061–2072. https://doi.org/10.1007/s10973-019-08046-6

[16] V.T. Jeielayaganga, M. Venkatesh, Tailoring the electrochemical properties of Ni-doped Co3O4 for advanced supercapacitor electrodes. Journal of the Indian Chemical Society, 102(11), (2025) 102170. https://doi.org/10.1016/j.jics.2025.102170

[17] T.Q. Tazim, M. Kawsar, M.S. Hossain, N.M. Bahadur, S. Ahmed, Hydrothermal synthesis of nano-metal oxides for structural modification: A review. Next Nanotechnology, 7, (2025) 100167. https://doi.org/10.1016/j.nxnano.2025.100167

[18] S.S. Krishnappa, S. Kalikeri, Calcination-tuned copper oxide nanoparticles for tomato seedling growth and vigour through nano-priming. Next Materials, 11, (2026) 101638. https://doi.org/10.1016/j.nxmate.2026.101638

[19] V.T. Jeielayaganga, M. Venkatesh, Influence of pH on the size and morphology of cobalt oxide nanostructures synthesized via microwave-assisted method. MRS Advances, 10(17), (2025) 2103–2111. https://doi.org/10.1557/s43580-025-01452-z

[20] C. Lin, Y. Wei, Enhanced reactivity of copper nanoparticles mass-produced by reductive precipitation in a rotating packed bed with blade packings. Journal of Materials Research and Technology, 9(6), (2020) 12328–12334. https://doi.org/10.1016/j.jmrt.2020.08.080

[21] M.A. Hessien, R.M. Khattab, H.E.H. Sadek, Synthesis and characterization of ZNO, MN3O4, and ZNMN2O4 spinel by new Chelation-Precipitation method: magnetic and antimicrobial properties. Journal of Inorganic and Organometallic Polymers and Materials, 35(5), (2024) 3739–3758. https://doi.org/10.1007/s10904-024-03489-3

[22] V. Molahalli, A. Sharma, K. Bijapur, G. Soman, A. Shetty, B. Sirichandana, B.G. M. Patel, N. Chattham, G. Hegde, Properties, synthesis, and characterization of CU-Based nanomaterials. In ACS symposium series, (2024) 1–33. https://doi.org/10.1021/bk-2024-1466.ch001

[23] R.M. Alhuthli, F.A. Alrahmany, B.a.A. Jahdaly, Electrocatalytic oxygen evolution on nano-CuOx modified electrodes: enhanced activity and stability through surface oxidation engineering. Results in Chemistry, 20, (2025) 103004. https://doi.org/10.1016/j.rechem.2025.103004

[24] J. Neiva, Z. Benzarti, S. Carvalho, S. Devesa, Green synthesis of CUO Nanoparticles—Structural, morphological, and dielectric characterization. Materials, 17(23), (2024) 5709. https://doi.org/10.3390/ma17235709

[25] Ratnawulan, A. Fauzi, S.H. Ae, Effect of calcination temperature on phase transformation and crystallite size of copper oxide (CuO) powders. AIP Conference Proceedings, 1868, (2017) 060009. https://doi.org/10.1063/1.4995173

[26] P.K. Kermanshahi, S. Estaji, E. Zivari, S. Moghari, P.A. Poshtahani, S. Moftakhari, H.A.

Khonakdar, A comprehensive review on electroactive MOF-reinforced nanocomposites: From material design to practical applications. Materials Today Sustainability, 32, (2025) 101229. https://doi.org/10.1016/j.mtsust.2025.101229

[27] U. Sidiqi, M. Ubaidullah, A. Kumar, D. Kumar, K. Muzammil, M. Imran, Progress on cupric oxide based nanomaterials: Exploring advancements in their synthesis, applications and prospects. Materials Science and Engineering B, 308, (2024) 117598. https://doi.org/10.1016/j.mseb.2024.117598

[28] X. Wang, K. Klingan, M. Klingenhof, T. Möller, J.F. De Araújo, I. Martens, A. Bagger, S. Jiang, J. Rossmeisl, H. Dau, P. Strasser, Morphology and mechanism of highly selective Cu(II) oxide nanosheet catalysts for carbon dioxide electroreduction. Nature Communications, 12(1), (2021) 794. https://doi.org/10.1038/s41467-021-20961-7

Author contributions

V.T. Jeielayaganga: Conceptualization, Methodology, data collection, analysis and interpretation of results, Writing Original manuscript, M. Venkatesh: Conceptualization, Formal analysis, Validation, Supervision, Review, Writing and Editing. All authors reviewed the results and approved the final version of the manuscript.

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Conflict of interest

The Author's declares that there is no conflict of interest anywhere.

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