

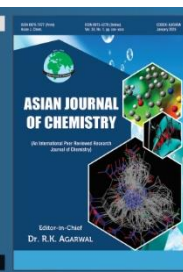


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Hydrothermal Synthesis of Chromium(II) Sulphate Doped Cuprous(I) Oxide of Optical Applications

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Chromium(II) sulphate (CrSO_4)-doped Cu_2O nanocomposites were synthesized through a simple, low-cost chemical method using CrSO_4 as a dopant to modify the structural, optical and electrochemical properties of Cu_2O . Four synthesis approaches were employed and the resulting materials were characterized with respect to morphology, particle size, crystal structure, optical absorption and electrochemical behavior. UV-Vis spectroscopy revealed a shift in the absorption edge after chromium incorporation, with the corresponding change in optical band gap suggesting modification of the electronic structure of Cu_2O . FTIR spectra exhibited the characteristic Cu–O vibrational band together with additional bands associated with interactions involving the dopant species, supporting the incorporation of Cr into the Cu_2O matrix. XRD patterns exhibited the characteristic reflections of cubic Cu_2O . Small changes in peak positions after Cr doping were consistent with lattice distortion caused by Cr-ion incorporation, while the cubic crystal structure was retained. SEM analysis revealed predominantly quasi-spherical particles with limited agglomeration and particle-size analysis placed the materials within the nanoscale range. Electrochemical measurements by cyclic voltammetry showed enhanced redox activity and improved charge-storage characteristics for the CrSO_4 -doped Cu_2O samples compared with undoped Cu_2O . Galvanostatic charge–discharge measurements yielded higher specific capacitance for the doped materials, together with improved cyclic reversibility and good cycling stability. These electrochemical characteristics support the potential application of CrSO_4 -doped Cu_2O nanocomposites as electrode materials for energy-storage systems. XPS analysis further established the elemental composition and chemical states of the synthesized CrSO_4 -doped Cu_2O nanoparticles. The combined structural, optical and electrochemical results demonstrate that controlled Cr incorporation can modify the physico-chemical properties of Cu_2O while preserving its cubic framework.

Keywords: Cyclic voltammetry, Cubic crystalline structure, Galvanostatic charge-discharge curves.

INTRODUCTION

Cuprous oxide (Cu_2O) is a p-type semiconductor with a direct band gap of approximately 2.0-2.2 eV. Its strong absorption in the visible region, relatively low toxicity and availability of constituent elements make it an attractive material for a range of technological applications. Cu_2O has been investigated for photocatalysis, solar-energy conversion, gas sensing and electrochemical energy storage [1-3]. Despite these favourable characteristics, pristine Cu_2O has limitations that can restrict its practical performance. Rapid charge-carrier recombination, moderate electrical conductivity and limited electrochemical activity can reduce its efficiency in opto-electronic and electrochemical systems. Modification of its physico-chemical properties through controlled doping is one approach used to address these limitations.

The incorporation of foreign cations, particularly transition metal ions, into the Cu_2O lattice can alter its electronic structure, defect concentration and surface characteristics. Such modifications can influence charge transport, catalytic behavior and electrochemical activity [4-7]. Chromium is of particular interest as a dopant due to its accessible oxidation states and ability to modify the local crystal environment. Incorporation of chromium species into Cu_2O may introduce lattice defects or localized electronic states, which can affect light absorption and charge-transfer processes. Chromium(II) sulphate (CrSO_4) can consequently serve as a dopant precursor for investigating the relationship between chromium incorporation and the structural, optical and electrochemical properties of Cu_2O .

The present work focuses on the synthesis and characterization of CrSO_4 -doped Cu_2O nanomaterials. Their structural

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and morphological characteristics were investigated using X-ray diffraction (XRD) and scanning electron microscopy (SEM), while UV-Vis absorption and Fourier-transform infrared (FTIR) spectroscopy were employed to examine their optical and vibrational characteristics. The electrochemical behaviour was assessed through cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) measurements to evaluate their charge-storage characteristics and suitability for electrochemical energy-storage applications [8-11]. Particle-size analysis was carried out to determine the nanoscale size distribution of the synthesized materials and assess changes associated with chromium incorporation. The study examines the influence of CrSO_4 doping on the structural, optical and electrochemical properties of Cu_2O , with emphasis on its potential application in electrochemical energy storage, catalysis and optoelectronic devices.

EXPERIMENTAL

Chromium(II) sulphate (CrSO_4), copper(II) sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), sodium hydroxide and glucose were used as the starting materials and reagents for the synthesis of CrSO_4 -doped Cu_2O nanomaterials. Deionized water was used for solution preparation and washing, while ethanol was used during the purification of the synthesized products. All chemicals were of analytical grade and were used without further purification. The required quantities of the reagents were calculated according to the desired precursor and chromium concentrations. Chromium(II) sulphate was added at concentrations corresponding to 1-5 wt.% Cr relative to the Cu_2O precursor system.

Characterization: The synthesized CrSO_4 -doped Cu_2O nanomaterials were characterized using an X-ray diffractometer (Bruker D8 Advance) with $\text{CuK}\alpha$ radiation to determine the crystalline phases, structural characteristics and crystallite size using the Scherrer equation. Fourier-transform infrared (FTIR) spectra were recorded using a Bruker Tensor 27 FTIR spectrometer over the range of $4000\text{--}500\text{ cm}^{-1}$ to identify characteristic Cu–O vibrations and other functional groups. Optical absorption characteristics were investigated using a Shimadzu UV-2600 UV-Visible spectrophotometer in the wavelength range of 200–600 nm and the optical band gap was estimated from the corresponding Tauc plot. Surface morphology was examined using a JEOL JSM-series scanning electron microscope (SEM) at different magnifications, while particle-size distribution was determined using a Malvern Mastersizer particle-size analyzer. Elemental composition and surface chemical states were analyzed using an X-ray photoelectron spectrometer (Thermo-Scientific K-Alpha) through survey and high-resolution spectra of Cu, Cr, O and S. Electrochemical properties were evaluated using a CHI-series electrochemical workstation/potentiostat-galvanostat through cyclic voltammetry (CV) over approximately 0.0–1.6 V and galvanostatic charge-discharge (GCD) measurements over 0.0–1.4 V at applied currents of 0.5–1.2 mA.

Hydrothermal synthesis of CrSO_4 -doped Cu_2O nanostructures: Chromium(II) sulphate-doped cuprous oxide ($\text{CrSO}_4\text{--Cu}_2\text{O}$) nanostructures were synthesized by a hydrothermal method. The procedure involved controlled precipi-

tation followed by hydrothermal treatment to obtain crystalline Cu_2O -based nanomaterials. In brief, 0.1 M $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ solution was prepared by dissolving the required amount of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 50 mL of deionized water under continuous magnetic stirring. An appropriate quantity of chromium(II) sulphate was then added to obtain the desired chromium concentration in the range of 1–5 wt.%. The mixture was stirred continuously to obtain a homogeneous solution. A 0.1 M NaOH solution was added dropwise under constant stirring until the pH reached approximately 10–12. Under these alkaline conditions, a blue precipitate of $\text{Cu}(\text{OH})_2$ was formed. After the precipitate settled, glucose was added gradually as a reducing agent under vigorous stirring. The glucose reduced $\text{Cu}(\text{II})$ species to $\text{Cu}(\text{I})$, promoting the formation of Cu_2O nuclei. The appearance of an orange-red colouration was taken as a visual indication of Cu_2O formation. The resulting suspension was transferred to a 100 mL Teflon-lined stainless-steel autoclave, which was sealed and maintained at $160\text{--}180\text{ }^\circ\text{C}$ for 6–12 h in a hot-air oven. The hydrothermal treatment facilitated crystal growth and was used to promote the incorporation of chromium species into the Cu_2O -based structure. After completion of the reaction, the autoclave was allowed to cool naturally to room temperature. The resulting precipitate was collected by centrifugation and washed several times with deionized water and ethanol to remove residual reagents and soluble impurities. The washed material was dried at $60\text{--}80\text{ }^\circ\text{C}$ for 6 h in a hot-air oven. The dried product was gently ground using an agate mortar to obtain $\text{CrSO}_4\text{--Cu}_2\text{O}$ nanopowder for subsequent structural, morphological, optical and electrochemical characterization.

RESULTS AND DISCUSSION

XRD analysis: The crystalline structure and phase composition of the $\text{CrSO}_4\text{--Cu}_2\text{O}$ nanomaterial were investigated by X-ray diffraction (XRD). The XRD pattern (Fig. 1) contains sharp and well-defined reflections, which confirmed the crystalline nature of the synthesized material. The most intense reflection at $2\theta = 36.668^\circ$ is assigned to the (111) plane of cubic Cu_2O . Other reflections at 29.435° , 42.067° , 61.321° , 73.367° and 77.569° are the characteristic of cubic Cu_2O and agree with the corresponding JCPDS reference data [12,13]. The reflections at 42.067° and 61.321° are indexed to the (200) and (220) planes, respectively. No additional distinct reflections associated with crystalline chromium-containing phases were detected within the measured diffraction range, suggesting that no separate crystalline Cr-based phase was formed at a detectable level.

The preservation of the characteristic Cu_2O diffraction pattern after CrSO_4 addition indicates that the cubic Cu_2O framework was maintained. Small changes in peak position and relative intensity may originate from lattice strain or local structural distortion associated with the presence of chromium species. No separate crystalline chromium containing phase was observed within the detection capability of the XRD measurement. Such a result can be associated with the low dopant concentration and/or dispersed nature of chromium in the Cu_2O matrix. Since XRD does not provide direct inform-

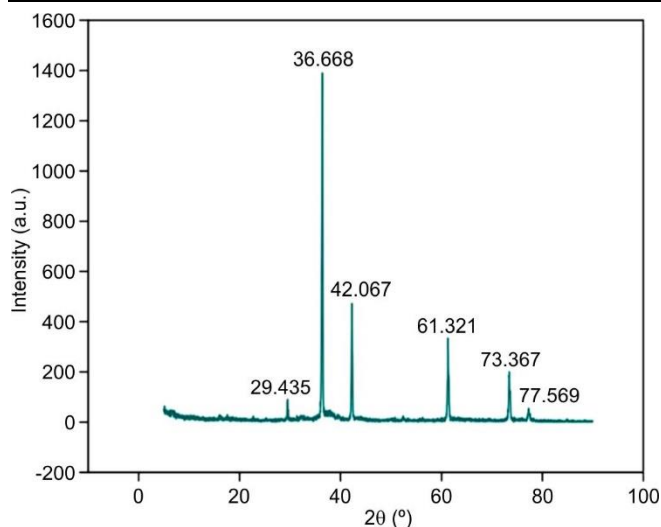


Fig. 1. XRD pattern of chromium(II) sulphate-doped cuprous oxide ($\text{CrSO}_4\text{-Cu}_2\text{O}$)

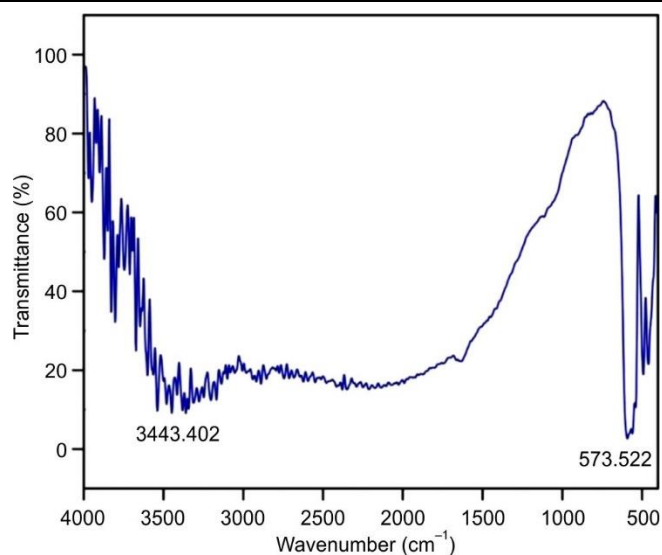


Fig. 2. FTIR spectrum of chromium(II) sulphate-doped cuprous oxide ($\text{CrSO}_4\text{-Cu}_2\text{O}$)

ation on the oxidation state or precise chemical environment of chromium, XPS analysis is required for further verification. The sharp and well-defined reflections are consistent with good crystallinity of the $\text{CrSO}_4\text{-Cu}_2\text{O}$ nanomaterial.

FT-IR analysis: The FTIR spectrum of the $\text{CrSO}_4\text{-Cu}_2\text{O}$ nanomaterial was recorded over the range of $4000\text{-}500\text{ cm}^{-1}$ to examine its characteristic vibrational bands and surface chemical groups. A broad absorption band at 3443.40 cm^{-1} is assigned to O-H stretching vibrations, which can arise from adsorbed moisture or hydroxyl groups associated with the particle surface. The band at 573.52 cm^{-1} is attributed to Cu-O vibrations and is consistent with the formation of the Cu_2O phase [14-16]. Weak bands observed in the $1200\text{-}1000\text{ cm}^{-1}$ region can be associated with S-O stretching vibrations of sulphate (SO_4^{2-}) ions originating from the CrSO_4 precursor (Fig. 2). Their presence suggests that a small amount of sulphate species may remain associated with the synthesized material. The absence of prominent absorption bands in the $2000\text{-}1500\text{ cm}^{-1}$ region suggests that significant organic residues were not retained after synthesis. The FTIR features, together with the XRD pattern, are consistent with the formation of Cu_2O and the presence of sulphate-related species.

UV-Visible analysis: The optical absorption characteristics of the CrSO_4 -doped Cu_2O nanoparticles were investigated by UV-Visible spectroscopy in the wavelength range of $200\text{-}600\text{ nm}$. The absorption spectrum exhibited distinct features at 208.65 , 237.62 and 289.05 nm . The band at 208.65 nm showed the highest absorption intensity, while the feature at 237.62 nm was comparatively weaker. A pronounced absorption band was observed at 289.05 nm , which correspond to a photon energy of approximately 4.29 eV (Fig. 3a). The absorption features in the ultraviolet region can be associated with electronic transitions within the $\text{CrSO}_4\text{-Cu}_2\text{O}$ system and may be influenced by lattice defects, dopant-related electronic states and changes in the local Cu-O environment.

The optical band gap was estimated from the Tauc plot by extrapolating the linear portion of the corresponding plot to the energy axis (Fig. 3b). The resulting band-gap value was approximately 4.3 eV . The close correspondence between the photon energy associated with the 289.05 nm absorption feature (4.29 eV) and the Tauc-derived value (4.3 eV) supports the assignment of the observed optical transition. The higher band-gap value relative to the commonly reported band gap

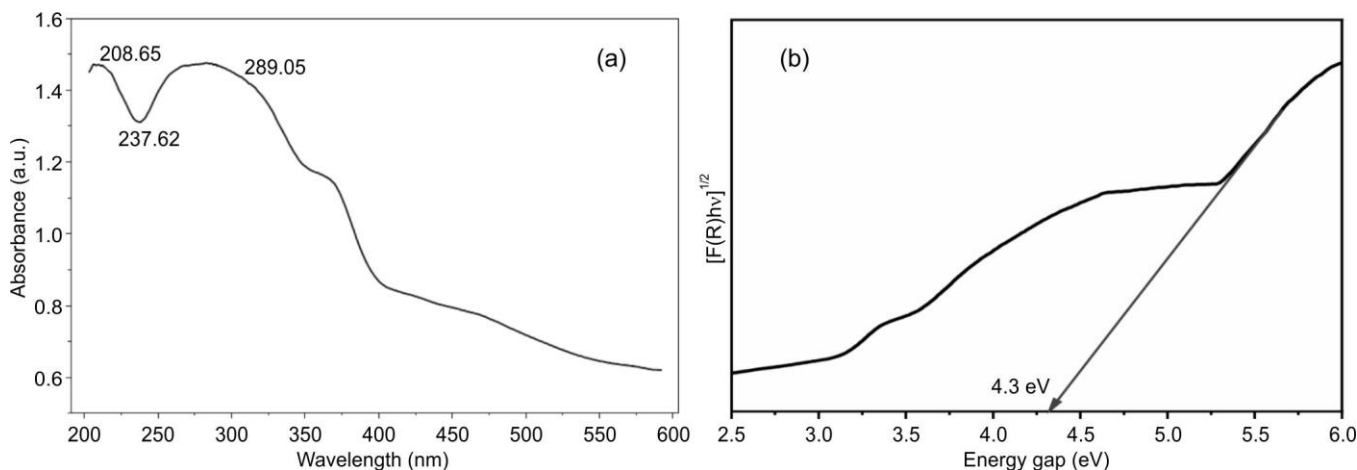


Fig. 3. (a) UV absorption spectrum of chromium(II) sulphate-doped cuprous oxide ($\text{CrSO}_4\text{-Cu}_2\text{O}$) and (b) Tauc plot

of bulk Cu_2O may arise from chromium incorporation, defect states, lattice distortion, particle-size effects or other changes in the electronic structure. Further analysis using undoped Cu_2O under identical experimental conditions would be required to isolate the specific contribution of CrSO_4 incorporation to the observed optical response.

SEM analysis: The surface morphology and particle characteristics of the CrSO_4 -doped Cu_2O nanomaterials were examined by SEM at different magnifications. The SEM images (Fig. 4) reveal variations in particle morphology, size, surface texture and degree of particle aggregation associated with the hydrothermal synthesis. At $2000\times$ and $5000\times$ magnification, the particles exhibit a relatively homogeneous distribution with comparable size and shape. Images recorded at $20,000\times$ and $35,000\times$ provide better detail of the particle surfaces and reveal a higher proportion of well-defined polyhedral features. The formation of these faceted structures can be related to the crystal growth process under hydrothermal conditions [17-19]. At $35,000\times$ magnification, closely packed crystalline particles and regions of secondary growth are visible, which may arise from nucleation and subsequent crystal growth during the hydrothermal treatment.

The particles exhibit relatively well-defined edges and compact morphologies, with no prominent surface cracks observed in the examined regions. Such features are consistent with the formation of structurally coherent Cu_2O particles. It should be observed that the particle-size range observed in the SEM images should not be referred to as the conventional

nanoparticle range when the measured dimensions are in the 200–800 nm region. Particles within this range are more appropriately, although they may consist of smaller primary crystallites.

XPS analysis: The XPS survey spectrum of the hydrothermally synthesized CrSO_4 - Cu_2O nanomaterial exhibited signals corresponding to Cu, Cr, O and S, thereby confirming the presence of these elements at the material surface (Fig. 5). The Cu and O signals are consistent with the Cu–O matrix of the Cu_2O -based material, while the Cr signal confirms the presence of chromium species associated with the synthesized sample. The S signal can be attributed to sulphate species originating from the CrSO_4 precursor. A carbon signal was observed, which can commonly arise from carbon adsorbed on the sample surface during handling and exposure to the atmosphere. A weak N signal was also similarly detected and may originate from trace surface contamination or residual precursor species. The survey spectrum establishes the elemental composition of the near-surface region and supports the presence of Cr- and S-containing species in the Cu_2O -based material.

Particle size analysis: Standard particle-size analysis was performed to evaluate the size distribution of the CrSO_4 - Cu_2O material. The differential and cumulative intensity distributions were plotted as a function of particle size to assess the distribution profile and size range of the measured particles (Fig. 6). The differential distribution curve (black line) displayed a single prominent mode between 200 and 300 nm, where the

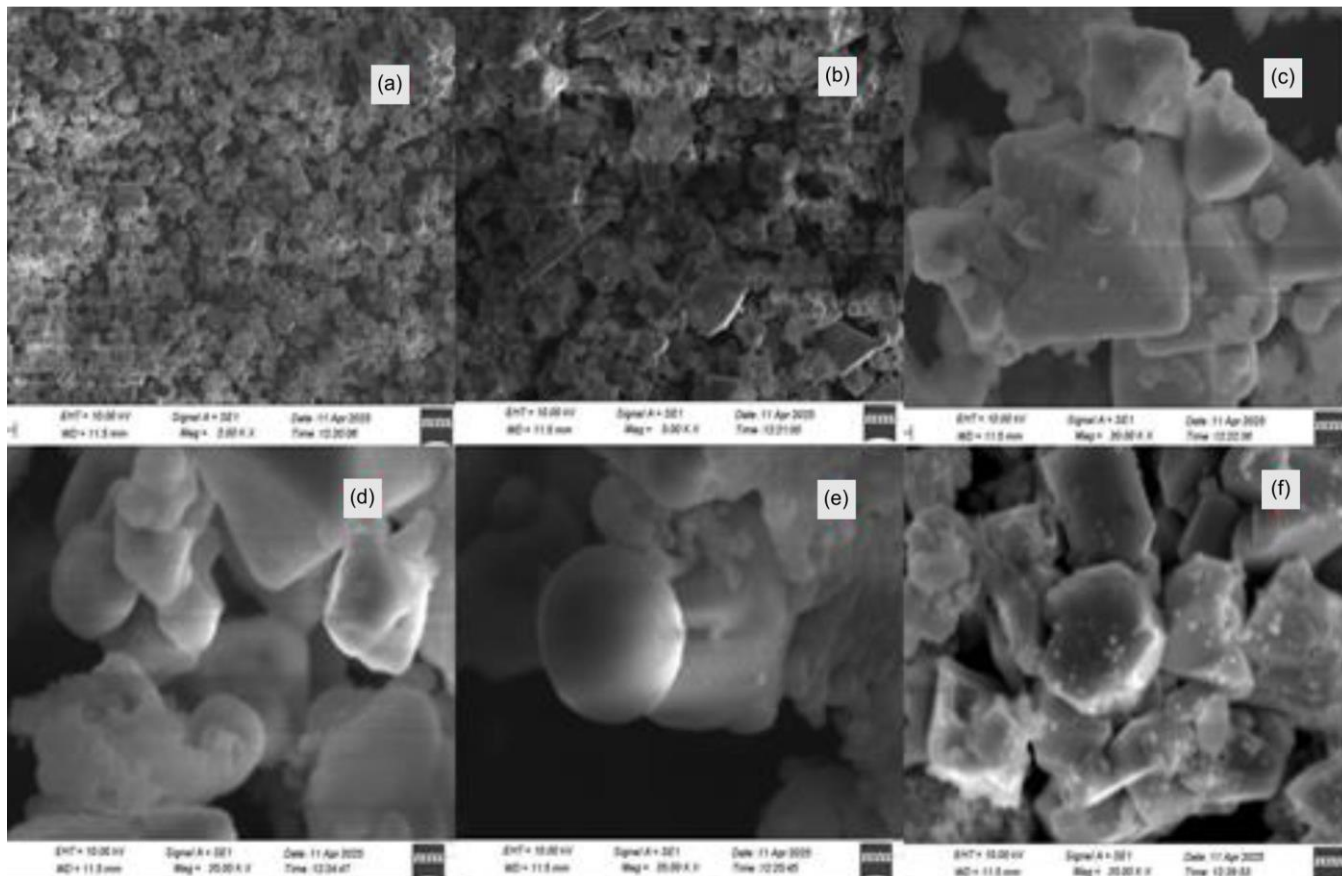
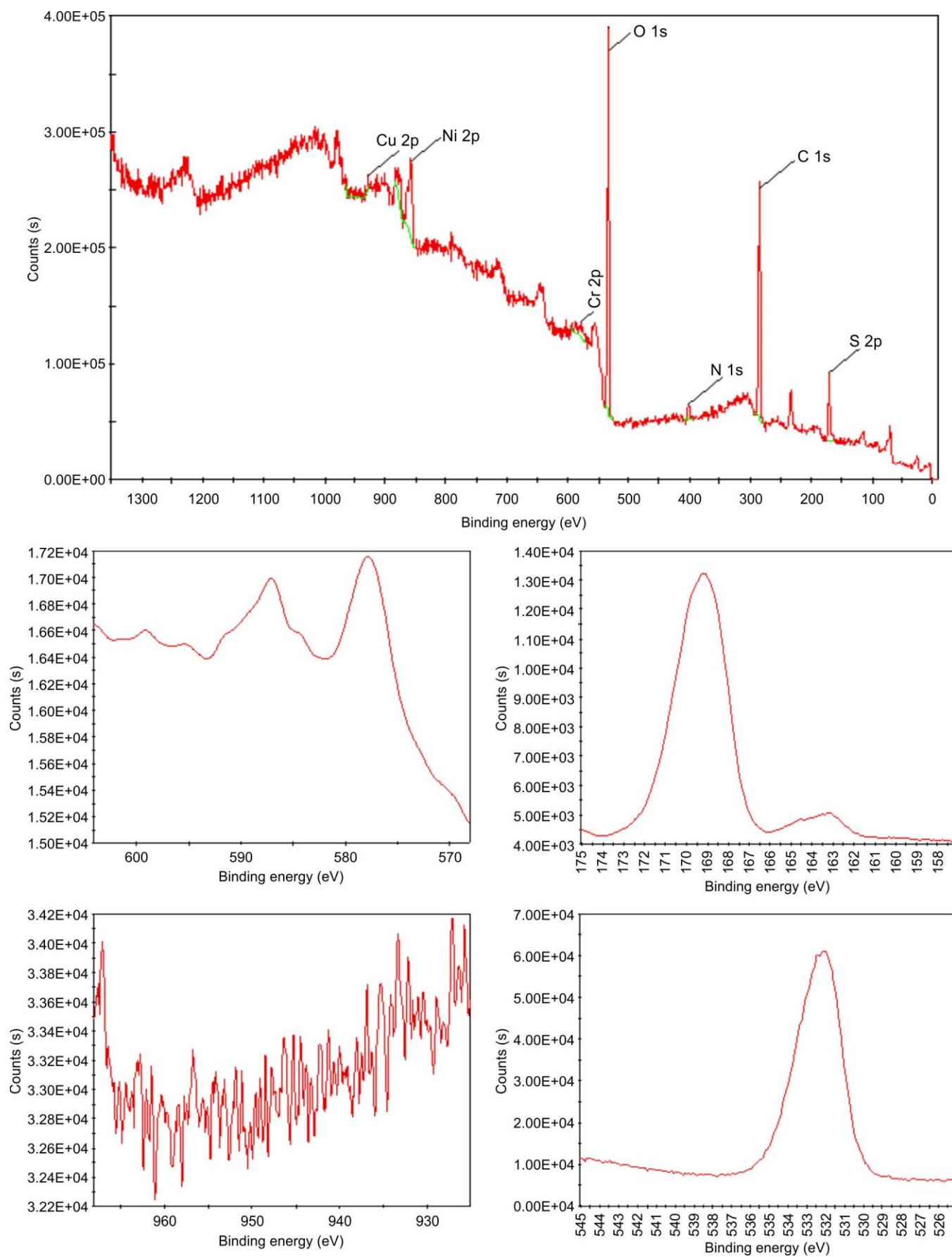


Fig. 4. SEM images of chromium(II) sulphate doped cuprous oxide (CrSO_4 - Cu_2O)

Fig. 5. XPS spectra of chromium(II) sulphate doped cuprous oxide ($\text{CrSO}_4\text{-Cu}_2\text{O}$)

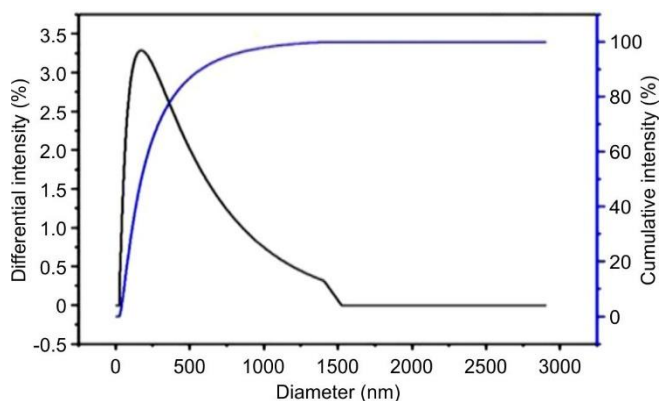


Fig. 6. Particle size distribution plots of chromium(II) sulphate doped cuprous oxide ($\text{CrSO}_4\text{-Cu}_2\text{O}$)

measured particle intensity was highest [20-23]. The intensity decreased progressively with increasing particle size and only a small fraction of the measured material extended beyond approximately 1500 nm. The intensity decreased to the negligible levels at larger particle sizes, with the limited high-size fraction potentially arising from particle aggregation or agglomeration.

The cumulative distribution curve (blue line) showed a rapid increase at lower particle sizes. Nearly 80-90% of the measured distribution was reached below 600 nm. The curve gradually approached 100% between 1000 and 1200 nm, which shows that the fraction of larger particles was relatively small. The results place the major fraction of the measured particles in the submicron range (Table-1). The measured particle size may be affected by particle aggregation and by the measurement technique.

TABLE-1
PARTICLE SIZE DISTRIBUTION DATA OF
 CrSO_4 -DOPED Cu_2O NANOPARTICLES

Particle diameter (nm)	Differential intensity (%)	Cumulative intensity (%)
100	1.2	15
200	3.2	45
400	2.5	75
600	1.8	88
800	1.2	95
1000	0.8	98
1400	0.3	100

Cyclic voltammetry (CV): The electrochemical behaviour of the CrSO_4 -doped Cu_2O electrode was investigated by cyclic voltammetry over a potential window of approximately 0.0-1.6 V at applied currents ranging from 10 to 100 mA. The CV profiles exhibited a gradual increase in current with increasing potential, followed by a pronounced current response near 1.3 V (Fig. 7). This feature may be associated with Faradaic oxidation processes involving surface species of the electrode, although assignment to a specific Cu or Cr redox couple requires further peak-resolved analysis. The absence of well-defined and sharp anodic and cathodic peaks suggests that the overall electrochemical response involves a combination of capacitive and Faradaic contributions rather than a single highly reversible redox process [24-27].

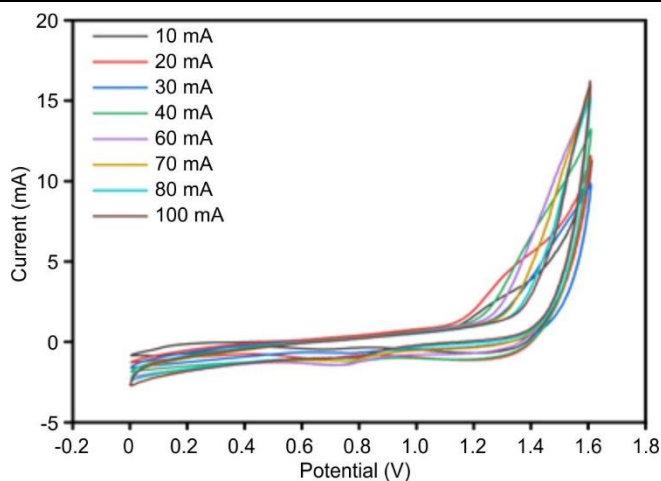


Fig. 7. Cyclic voltammograms of chromium(II) sulphate-doped cuprous oxide ($\text{CrSO}_4\text{-Cu}_2\text{O}$)

The CV profiles remained broadly comparable across the applied conditions, with only minor changes in shape at higher current levels. These deviations may be associated with ohmic resistance, restricted ion transport or polarization effects within the electrode. The contribution of chromium to the electrochemical response may be associated with changes in the electronic structure and surface chemistry of the Cu_2O matrix. The combined capacitive and Faradaic characteristics observed in the CV profiles indicate pseudocapacitive behaviour of the $\text{CrSO}_4\text{-Cu}_2\text{O}$ electrode.

GCD analysis: Galvanostatic charge-discharge (GCD) measurements of the $\text{CrSO}_4\text{-Cu}_2\text{O}$ electrode were performed at applied currents ranging from 0.5 to 1.2 mA within a potential window of approximately 0.0-1.4 V. The GCD profiles (Fig. 8) exhibited broadly symmetrical charge and discharge characteristics across the investigated current range. This profile is consistent with a reversible charge-storage response. The quantitative assessment of Coulombic efficiency requires calculation from the corresponding charge and discharge times.

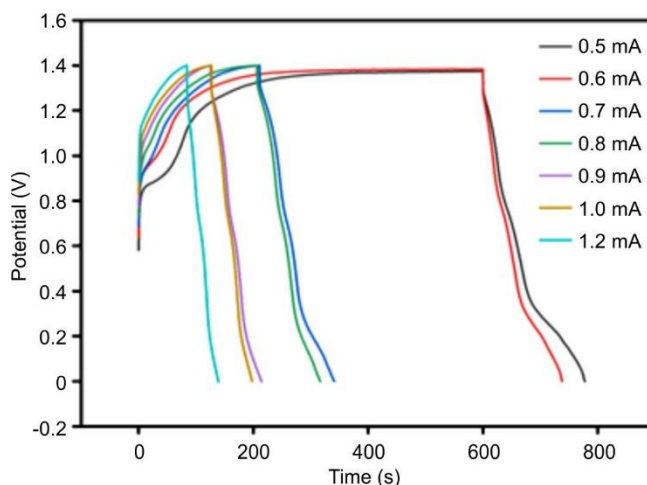


Fig. 8. GCD curves of chromium(II) sulphate doped cuprous oxide ($\text{CrSO}_4\text{-Cu}_2\text{O}$)

The charge curves showed an initial potential change that can be associated with the internal resistance of the electrode and the electrochemical cell. The subsequent region exhibited

a relatively gradual and quasi-linear potential variation. This behaviour suggests the contribution of both electric double-layer capacitance and Faradaic charge-storage processes. A deviation from an ideal triangular profile was observed near the upper potential region at approximately 1.3-1.4 V. This feature may be associated with Faradaic reactions involving surface or near-surface species.

The charge and discharge times decreased with increasing applied current. This behaviour is expected at higher current since the electrode reaches the selected potential within a shorter time and ion transport becomes more limited during rapid charging and discharging [28,29]. The GCD profiles retained similar characteristics across the investigated current range although some deviation from the ideal triangular shape was observed at higher currents. Such changes may arise from increased ohmic losses and electrode polarization under high current conditions. The longer discharge time observed at lower applied current is consistent with higher charge storage capacity under the corresponding measurement conditions. The relatively smooth charge-discharge profiles suggest a stable electrochemical response of the electrode. The observed GCD characteristics are consistent with mixed capacitive and Faradaic charge-storage behaviour and provide support for the use of CrSO₄-doped Cu₂O in electrochemical energy storage applications.

Conclusion

In this study CrSO₄-doped Cu₂O nanomaterials were synthesized successfully by a simple hydrothermal method. XRD analysis confirmed the formation of crystalline cubic CrSO₄-Cu₂O with no distinct crystalline chromium containing secondary phase detected within the measured diffraction range. FTIR analysis identified Cu–O vibrations together with O–H and sulphate-related bands. XPS survey analysis confirmed the presence of Cu, Cr, O and S on the material surface. The UV-Vis results showed characteristic optical absorption features with a Tauc-derived optical band gap of approximately 4.3 eV. SEM analysis revealed well-defined faceted particles with some degree of particle aggregation. Particle-size analysis showed a dominant distribution in the 200-300 nm range with approximately 80-90% of the measured distribution below 600 nm. The electrochemical measurements showed mixed capacitive and Faradaic charge-storage characteristics. The GCD profiles exhibited relatively symmetrical charge-discharge behaviour with changes in discharge time as the applied current increased. These results provide preliminary evidence for the electrochemical activity of CrSO₄-Cu₂O and its potential use in energy-storage applications.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI.

All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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