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HYDROTHERMAL SYNTHESIS AND ELECTROCHEMICAL PERFORMANCE OF HEXAGONAL CuS NANOSTRUCTURES FOR SUPERCAPACITOR APPLICATIONS

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ABSTRACT

Recently, copper sulfide (CuS) has emerged as a promising material with attractive electrochemical properties for potential energy-storage applications. The hexagonal CuS nanostructures were synthesised successfully using a hydrothermal method at 150 ℃ without adding surfactants. The synthesised material was characterised by Fourier transform infrared spectroscopy (FTIR), field-emission scanning electron microscopy (FESEM), and powder X-ray diffraction (PXRD) to investigate its structural and morphological characteristics. The results confirm the formation of highly crystalline hexagonal CuS using PXRD analysis, while Cu-S bonding is demonstrated in FTIR analysis. The FESEM images have clearly revealed a well-developed hexagonal nanostructure of the material, and UV-Visible spectroscopy demonstrated the optical absorption behaviour of the synthesised CuS material. Furthermore, cyclic voltammetry measurements indicate the electrochemical activity of the CuS material, demonstrating its potential as an electrode material for electrochemical energy storage. This simple method of synthesising materials, combined with favourable structural characteristics, highlights their potential for the development of cost-effective energy-storage devices. This study contributes to Sustainable Development Goal (SDG) 7: Affordable and Clean Energy through the development of CuS-based electrode materials for potential electrochemical energy-storage applications. The favourable structural and electrochemical characteristics of the synthesised CuS nanostructures demonstrate their potential as electrode materials for future electrochemical energy-storage systems. The present findings provide a basis for further investigation of CuS-based electrodes with improved energy-storage performance.

Keywords: Copper Sulfide, Electrochemical Properties, Energy-Storage, Hexagonal Nanostructures, Hydrothermal Synthesis.

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INTRODUCTION

Nanoscience and nanotechnology have emerged rapidly because of the special chemical, physical and optical characteristics exhibited by materials at the nanoscale. This has become one of the most promising disciplines that is developing rapidly. The materials are engineered at the molecular level and range from one to several nanometres in size. At the nanoscale, materials exhibit distinct physicochemical characteristics arising from their reduced dimensions and large surface area. These features can enhance their surface reactivity and quantum effects, making them attractive for diverse technological applications. CuS nanostructures have been prepared using several synthetic approaches, which include microwave-assisted synthesis, solution-based methods, template-assisted synthesis, hydrothermal and solvothermal techniques, polyol methods, solid-state synthesis, chemical vapour deposition, wet chemical methods, and sonochemical synthesis.[1-10] Among these methods, hydrothermal synthesis has been extensively studied due to its simple, low-cost and environmentally friendly route to prepare highly crystalline nanomaterials with tunable shape under relatively mild reaction conditions. Copper is a good electrical and thermal conductor. It is also corrosion-resistant, nonmagnetic, antimicrobial, recyclable, and catalytic and saves

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electrical power when wound in a coil. Recent research has explored its applications in dielectrics, optics, biomedicine, and bioscience. CuS is a p-type semiconductor formed by a combination of group I-VI elements. It exhibits exceptional electrical, optical, catalytic and electrochemical properties at the nanoscale. CuS has been extensively studied for applications in solar cells, photocatalysis, optical filters, superconductors, thermoelectric conductors, cooling applications, and as an ideal material for solar energy applications.[11-15] Copper sulfide has attracted significant attention in recent years because of its fascinating range of nanostructures, including nanospheres, nanowires, nanoplates, nanorods, flower-shaped architectures, and nanoribbons.[16] Furthermore, CuS can be synthesised in many morphologies, such as nanospheres, nanorods, nanowires, nanoplates, nanoflowers, and nanoribbons, which all play a significant role in influencing its optical and electrochemical performance.[17,18,19] These unique morphologies contribute to its versatile properties and broaden its potential applications in various fields. Although numerous studies have reported different synthesis strategies for producing CuS with controlled shapes and sizes, the underlying formation mechanism and growth chemistry of CuS are still not fully understood. Some challenges still remain despite the significant progress in controlling the morphology and size of CuS nanostructures. Many of the described synthesis routes rely on surfactants or templates, or require complicated reaction conditions, which increase the overall processing cost and can introduce impurities that affect the material's performance.[20,21,22] Nevertheless, the growth mechanism for the preparation of phase-pure hexagonal CuS with adjustable morphology through a simple hydrothermal process is still incompletely clarified. In particular, a simple additive-free synthesis route that produces well-defined hexagonal CuS nanostructures while retaining favourable electrochemical characteristics remains of practical interest for energy- storage applications. Thus, there is still an important research challenge to develop a facile and additive-free hydrothermal strategy to prepare highly crystallised CuS nanostructures with desirable electrochemical properties. In hydrothermal processing, Nanophase materials are formed through chemical reactions in an aqueous or organic environment under elevated temperature and pressure, typically in the presence of an alkali. Pressure and temperature affect the basic properties of the solvent.[23] The autoclave is heated up in such a way that there are two temperature levels. The nutrients are dissolved at the hot level, and the saturated aqueous solution at the low level is transferred to the high level by means of the convective flow of the solution. The cold and denser solution at the high level moves downward as the flow of the solution increases. The solution becomes supersaturated at the high level, and as the temperature decreases, nucleation takes place, forming nanocrystals. The present study aims to synthesise hexagonal CuS nanostructures using a straightforward hydrothermal method without employing templates or surfactants. It also investigates their structural, optical, morphological, and electrochemical properties. The synthesized CuS nanostructures were characterized by PXRD, FTIR, UV-Visible spectroscopy, FESEM, and cyclic voltammetry to assess their potential for electrochemical Energy-storage applications.

The development of CuS-based electrode materials through a simple and cost-effective synthesis approach is relevant to Sustainable Development Goal (SDG) 7: Affordable and Clean Energy, particularly through its potential contribution to sustainable electrochemical Energy-storage applications. The successful synthesis of CuS hexagonal plates with an average edge length of 1 μm, thickness of 100 nm, and crystallite size of around 45 ± 2 nm (according to Scherrer estimation) through an assisted agent-free hydrothermal technique was achieved.[24] In the case of a proper Cu:S molar ratio, it was demonstrated that the synthesis of covellite (CuS) could be achieved without regard to the temperature at which the reaction took place.[25] Nevertheless, several impurities were discovered along with CuS in cases where the reaction temperature did not suffice to break down the excess crystallites. Moreover, it was established that the Cu:S molar ratio was very significant for controlling the level of cyclooctasulfur (S8) present in the resulting powder product. Textile and leather industry dyes are a major source of water pollution. Conventional methods for treating textile and leather dye wastewater are ineffective and can produce harmful by-products. Photocatalysis is a promising new approach for degrading dyes in wastewater. Copper sulfide (CuS) is a promising photocatalyst for this application due to its band gap, low toxicity, and porosity. This study also investigated the degradation of crystal violet dye under visible light using a prepared CuS material.[26,27,22]

EXPERIMENTAL

Materials and Synthesis of CuS Nanostructures

All chemicals were of analytical grade and used without any further purification. Throughout the study, distilled water was used for synthesis. First, copper nitrate trihydrate (0.02 mol) was dissolved in 20 mL of distilled water using a magnetic stirrer, resulting in a blue solution. Similarly, thiourea (0.05 mol) was slowly added, and the blue solution turned green upon continuous stirring. The green solution changed to pale green and then to pale white. The solution was transferred into a Teflon-lined stainless steel autoclave and gently sealed. The sample was left in the furnace for 24 hours at 150 °C and then cooled to room temperature. Blue-black precipitation occurred, which was recovered by filtration. The material was rinsed with ethanol and distilled water several times and finally dried at 500 °C. The same procedure was repeated, but the reaction time was kept at 5 hours instead of 24 hours to study the growth of the CuS nanoparticles and their morphological structure. The chemical reaction of copper nitrate and thiourea is given below:

$$(Cu\ (NO_3)2.3H_2O) + (CS\ (NH_2)) \rightarrow CuS \downarrow + CO \uparrow + 4NO_2 \uparrow + 5H_2 \uparrow$$

Characterisation of CuS NPs

The characteristics and phase purity of the synthesised CuS nanoparticles were analysed. X-ray diffraction (XRD) was used to study the phase purity and crystal structure of the synthesized CuS nanostructures. The surface morphology and microstructural features were investigated using field-emission scanning electron microscopy (FESEM). The optical properties of the synthesized CuS nanostructures were characterized using ultraviolet-visible (UV-Vis) spectroscopy (Shimadzu 1800). The photoluminescence (PL) spectra of the CuS nanoparticles were recorded using a fluorescence spectrophotometer (Shimadzu RF-6000).

RESULTS AND DISCUSSION

UV-Vis Analysis

A detailed study was carried out on the synthesized CuS nanostructures to better understand their growth behavior and formation mechanism under different experimental conditions. In addition, the optical characteristics of the prepared CuS structures were examined using UV-Visible absorption spectroscopy. The UV-Vis absorption spectrum of the as-prepared CuS nanostructures is shown in Fig. 1. The absorption peak was observed around 282 nm. The observed absorption feature reflects the optical response of the synthesized CuS nanostructures and supports the formation of the semiconductor phase. This optical behavior is consistent with the reported characteristics of CuS nanostructures and provides additional evidence for the successful synthesis of the material.[28] The absorption behavior correlates with the electronic transition taking place in the CuS nanostructures and shows the successful formation of the semiconductor phase. The observed absorption behavior confirms the successful formation of CuS nanostructures and is in accordance with the characteristic optical response exhibited by hydrothermally synthesized CuS materials. The optical absorption properties reveal that the synthesized CuS nanostructures are suitable for electrochemical and energy-related applications.

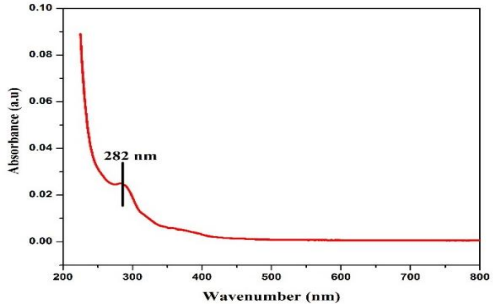


Fig.-1: UV-Vis Absorption Spectrum of the as-prepared CuS Nanostructures

FT-IR Analysis

FT-IR spectroscopy was carried out to identify the functional groups involved in the synthesis of CuS nanoparticles. The FTIR bands observed confirm the successful formation of CuS nanostructures and

indicate the presence of characteristic Cu-S bonding and surface functional groups. These surface groups could potentially be contributing to the stability and electrochemical behaviour of the synthesized material. The spectrum shown in Fig.-2 displayed several characteristic peaks corresponding to different functional groups. The peak at 713 cm-1 was assigned to the S-H stretching vibration of thiolate groups, while peaks at 1015 and 1044 cm-1 correspond to S-S stretching vibrations of disulfide groups. The peak at 1568 cm-1 confirmed the Cu-S bond formation, indicating the successful synthesis of copper sulfide nanoparticles. In addition, the peaks observed at 2977, 3206, 3304, and 3420 cm-1 were attributed to O-H, C=O, and N-H functional groups.[28] These functional groups may contribute to the reduction, stabilization, and capping of the synthesized CuS nanoparticles.

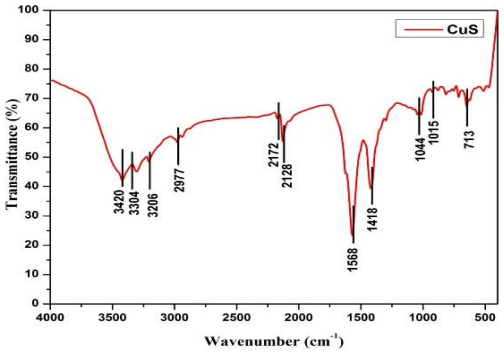


Fig.-2: FT-IR Spectra of the as-prepared CuS Nanostructures Showing the Characteristic Vibrational Bands

The identification of these functional groups indicates that the synthesized CuS nanoparticles contain the functional groups of thiosulfate, disulfide, and amine. The presence of these functional groups confirms the successful synthesis of CuS nanostructures and indicates the potential that the residual surface species may be involved in the stability and electrochemical behavior of the synthesized material. It is important to note that the functionality of CuS NPs depends upon the presence of these functional groups, which contribute to the catalytic activity, sensing capacity, and optoelectronic properties of CuS nanoparticles. Apart from the functional groups mentioned above, the FT-IR spectrum also exhibits a broad band in the range of 3400-3600 cm^{-1}. This broad band is assigned to the vibration of O-H groups. It is possible that water molecules may be present on the surface of CuS nanoparticles because of the synthesis process used in this study. The observed FTIR features are consistent with those reported for hydrothermally synthesized CuS nanostructures in earlier studies.

XRD Analysis

The analysis of the synthesized copper sulfide (CuS) nanoparticles through powder X-ray diffraction (XRD) has yielded significant insights into their phase structure, crystallinity, and particle size. The XRD pattern in Figure 3 shows a distinct series of well-defined peaks, each occurring at a specific 2θ angle: 27.56, 29.41, 31.90, 32.72, 38.94, 44.06, 47.97, 52.73, 56.04, 59.35, 63.58, 67.17, 73.96, 79.12, and 89.21. This pattern of peaks is a characteristic of crystalline materials, indicating the ordered arrangement of atoms within the CuS nanoparticles. The excellent correspondence between the observed peak positions in the experimental XRD pattern and the reference XRD pattern found in JCPDS card file No. 79-2321 is particularly noteworthy.[29] This card file represents the standard XRD pattern for CuS, and the agreement between the observed peaks and those in the reference pattern confirms that the synthesized CuS nanoparticles indeed possess the expected hexagonal crystal structure. The absence of other diffraction peaks shows that no other detectable secondary crystalline phases were generated during synthesis, reflecting the high phase purity of the synthesised CuS nanostructures. The appearance of these sharp peaks and their alignment with the reference pattern underscore the purity of the synthesised CuS nanoparticles. This implies that the nanoparticles are predominantly composed of CuS material, without the presence of detectable impurities or secondary phases that might distort the XRD pattern.In conclusion, the powder X-ray diffraction analysis yielded a comprehensive understanding of the synthesised CuS nanoparticles. The series of sharp and well-defined peaks, in agreement with the reference pattern, confirms their high crystallinity and hexagonal crystal structure.

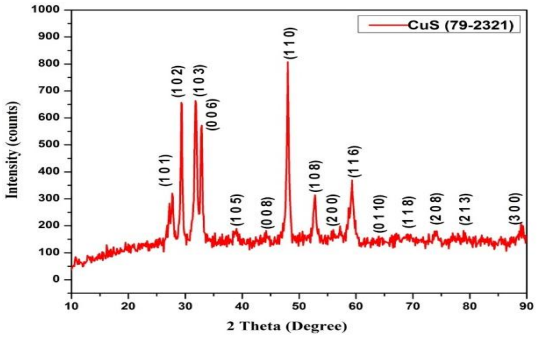


Fig.-3: Powder X-ray Diffraction (XRD) Pattern of the as-prepared CuS Nanostructures Confirming the Hexagonal Crystalline Phase

The absence of impurity peaks reaffirms their purity. The broadening of peaks, a characteristic of nanoscale materials, provides a basis for estimating the average particle size. Overall, this analysis not only validates the successful synthesis of CuS nanoparticles but also provides essential information about their structural properties, paving the way for informed applications and further research.

FESEM Analysis

Field emission scanning electron microscopy (FESEM) analysis revealed the hexagonal morphology of covellite (CuS) nanoparticles. The morphological images (as shown in Fig.-4 (a, b and c)) show that the CuS nanoparticles are well aligned and have a hexagonal plate-like shape. The average particle size of the CuS nanoparticles was approximately 100 nm. The hexagonal morphology of CuS nanoparticles is attributed to the crystal structure of covellite. Covellite is a hexagonal crystal system with a space group of P63/mmc. This crystal structure results in the formation of hexagonal plate-like nanoparticles.

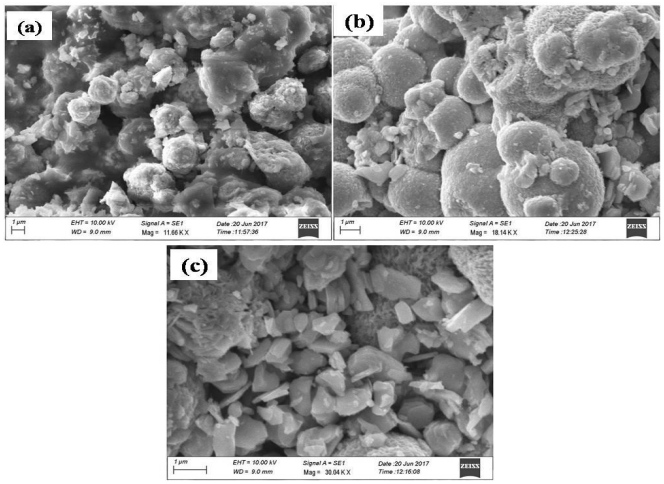


Fig.-4: FESEM Images of the as-prepared CuS Nanostructures Showing their Hexagonal Plate-Like Morphology

Electrochemical Analysis

The reduction of the CuS nanoparticles in the aqueous solution by means of the cyclic voltammograms is shown in Fig.-5. The initial cathodic peak around -0.9 V relates to the reduction of the CuS nanoparticle to Cu2S. The second cathodic peak around -1.5 V indicates the formation of copper from Cu2S. The anodic peak at approximately 0.5 V relates to the oxidation of copper back to CuS. It should be noted that the width of the anodic peak represents the oxidation rate of Cu back to CuS.[30] The structural and electrochemical characteristics observed for the synthesised CuS nanostructures demonstrate their potential for application in electrochemical energy-storage systems. In this context, the present findings are relevant to Sustainable Development Goal (SDG) 7: Affordable and clean energy, by contributing to the development of candidate electrode materials for sustainable energy-storage technologies.

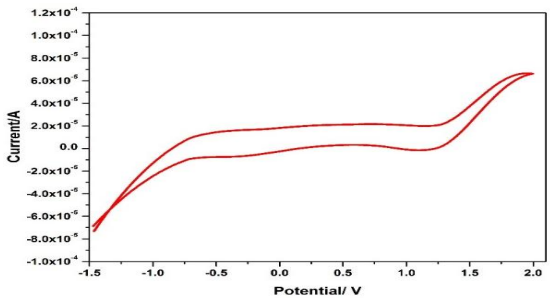


Fig.-5: Cyclic Voltammetry of the as-prepared CuS Nanostructures Showing Their Electrochemical Redox Behaviour

CONCLUSION

This research work presents a facile yet effective method for synthesising CuS nanostructures as an electrode material for supercapacitors. The electrochemical behaviour of the CuS electrodes in supercapacitors can be explained by the ability of the materials to facilitate the transport rates of electrons and electrolyte ions during charge/discharge processes. As a result, the capacitive behaviour improved. The development of CuS-based electrode materials through a simple hydrothermal route is relevant to SDG 7 (Affordable and Clean Energy) storage technologies. Further studies involving specific capacitance, cycling stability, rate capability, and electrode optimisation would be useful to establish the practical energy-storage performance of the CuS nanostructures.

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CONFLICT OF INTERESTS

The authors declare that they have no conflicts of interest regarding the publication of this manuscript.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-7: Affordable and Clean Energy*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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