iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 12 **Issue:** VI **Month of publication:** June 2024

DOI: https://doi.org/10.22214/ijraset.2024.62599

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International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
Volume 12 Issue VI June 2024- Available at www.ijraset.com

Preparation and Characterization of $CuFeS_2$ Nanoparticles Synthesized via Hydrothermal Method

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***Abstract:** This research involved the successful synthesis of $CuFeS_2$ nanoparticles using a hydrothermal method. The $CuFeS_2$ nanoparticles were characterized using various analytical techniques, including X-ray diffraction (XRD), Raman spectroscopy, field emission scanning electron microscopy (FESEM), energy-dispersive X-ray spectroscopy (EDX), UV-Visible spectroscopy, and Brunauer-Emmett-Teller (BET) surface area analysis. The tetragonal crystalline structure was determined using X-ray diffraction (XRD) examination, indicating a particle size of 14 nm. The existence of chalcopyrite $CuFeS_2$ nanoparticles was established by the analysis of Raman spectra. The amorphous morphology was observed by FESEM imaging. The existence of Cu, Fe,and S elements was verified using EDX analysis, with no significant impurities observed. The UV-Visible investigation revealed a significant capacity for absorption within the wavelength region of 500-600 nm, accompanied by an energy band gap of 2.35 eV. The examination of BETsurface area revealed a surface area of 62 m^2/g and a pore size of 10 nm. The obtained findings suggest that the $CuFeS_2$ nanoparticles possess favorable properties that render them appropriate for use in photocatalytic applications.*

***Keywords:** $CuFeS_2$, Chalcogenides, Hydrothermal Method, Characterization analysis.*

I. INTRODUCTION

Nanomaterials have attracted considerable interest in recent years owing to their distinctive characteristics and possible uses in industries including electronics, catalysis, sensing, and environmental remediation [1-2]. Transition metal chalcogenides have garnered significant attention as potential nanomaterials due to their unique electrical, optical, and catalytic characteristics [3-5]. Chalcopyrite copper iron sulfide ($CuFeS_2$) is a semiconductor compound that belongs to the I-III-VI_2 ternary group. The material has distinctive characteristics, includinga high Neel temperature and outstanding electrical and optical capabilities, characterized by a very narrow optical band gap [6]. $CuFeS_2$ sometimes referred to as chalcopyrite, is a ternary compound that has intriguing properties such as a small bandgap, high absorption coefficient, and exceptional chemical stability. $CuFeS_2$ nanoparticles possess these characteristics that renderthem very appealing for use in solar cells, photocatalysis, and photovoltaic systems [7].

Nevertheless, the achievement of successful synthesis and comprehensive characterization of $CuFeS_2$ nanoparticles is crucial in order to fully harness their capabilities in many technological domains. Within this particular context, the hydrothermal approach emerges as a very adaptable and efficient methodology for the production of nanoparticles, exhibiting meticulous control overtheir dimensions, structure, and crystalline properties [8]. The characteristics of the resultant nanoparticles may be customized to match particular needs by manipulating reaction parameters, including temperature, pressure, and precursor concentrations. Several synthesis methods have been explored to fabricate $CuFeS_2$ nanoparticles, each offering distinct advantages and challenges. The synthesis method involves the controlled hydrothermal reaction of copper, iron, and sulfur precursors to generate nanoparticles that are uniform and well-defined. Among these methods, the hydrothermal synthesis route has gained significant attention for its ability to produce nanoparticles with controlled size, morphology, and crystallinity. The hydrothermal method involves the reaction of precursor materials under elevated temperature and pressure conditions in an aqueous solution, facilitating the nucleation and growth of $CuFeS_2$ nanoparticles with tailored properties [9-10].

Understanding the structural and optical properties of $CuFeS_2$ nanoparticles is crucial for optimizing their synthesis and harnessing their potential in various applications. The crystal structure of $CuFeS_2$, characterized by a tetragonal lattice arrangement, influences its electronic and optical behaviors [11].

Additionally, the bandgap of $CuFeS_2$ can be tuned by adjusting its composition and morphology, enabling tailored absorption properties for specific applications. The optical properties of $CuFeS_2$, including its absorption and emission spectra, play a pivotal role in applications such as photovoltaics, photocatalysis, and sensing. $CuFeS_2$ nanoparticles exhibit unique optical properties arising from quantum confinement effects and surface plasmon resonance phenomena, which can be exploited for efficient light harvesting and charge separation in photovoltaic devices, as well as for enhancing catalytic activity in photocatalytic reactions [12]. Furthermore, $CuFeS_2$ nanoparticles have garnered attention for their promising applications in various fields, including solar cells, photoelectrochemical devices, gas sensors, and biomedical imaging. The tunable bandgap and high absorption coefficients of $CuFeS_2$ make it an ideal candidate for photovoltaic applications, while its catalytic activity and biocompatibility render it suitable for sensing and biomedical applications [13].

In this study, we embark on a comprehensive exploration of $CuFeS_2$ nanoparticles fabricated through the hydrothermal method, delving into their production, analysis, and prospective applications. Through meticulous synthesis processes, we successfully generate $CuFeS_2$ nanoparticles, followed by an exhaustive evaluation employing a diverse array of analytical techniques. Our findings underscore the remarkable potential of these nanoparticles across various technological domains, with particular emphasis on their applicability in photocatalysis.

II. EXPERIMENTAL SECTION

The hydrothermal method was employed for the facile synthesis of $CuFeS_2$ particles. Initially, copper chloride (1 mol; $CuCl_2$; Sigma–Aldrich) and ferric chloride (1 mol; $FeCl_36H_2O$; Sigma– Aldrich) were dissolved in 100 mL of pure water under continuous stirring for duration of 60 minutes. Following this, thiourea (1 mol, $(NH_2)_2CS$, Sigma–Aldrich) was gradually added to the CuFe solution, reaching a volume of 100 ml. The resulting $CuFeS_2$ solution was then transferred into a Teflon-lined stainless steel autoclave and maintained at a temperature of 180°C for a period of 12 hours. After the hydrothermal reaction, the obtained powders underwent filtration and were thoroughly washed with hot distilled water before undergoing vacuum drying. The residual substance was collected, washed with deionized water, and subsequently incubated overnight at 45°C to ensure complete drying. Additionally, a schematic representation of the experimental setup is provided in Figure 1.

The synthesized $CuFeS_2$ nanoparticles underwent comprehensive characterization utilizing state- of-the-art analytical techniques to elucidate their structural, morphological, elemental composition, surface area, and optical properties. Structural analysis was performed using a high-resolution X-ray diffractometer (XRD) (XPERT-PRO) to determine the crystalline structure, while Raman spectroscopy (WITech CRM200) provided insights into the molecular vibrational modes. Morphological and elemental composition analysis was carried out using a Field Emission Scanning Electron Microscope (FESEM) (Sigma HV – Carl ZEISS) equipped with Bruker Quantax 200-Z10 Energy-Dispersive X-ray Spectroscopy (EDS) detector. The surface area of the nanoparticles was determined using a Nova 2200e Analyzer through N_2 adsorption and desorption processes. Optical properties were investigated using UV-Vis spectrometer (Hitachi-UH5300, λ = 200–900 nm) to assess their absorbance characteristics in the ultraviolet-visible range. These characterization techniques collectively provided a comprehensive understanding of the synthesized $CuFeS_2$ nanoparticles, paving the way for their potential applications in various technological fields.

III. RESULTS AND DISCUSSION

Figure 2(A) presents the X-ray diffraction (XRD) analysis results of the synthesized $CuFeS_2$ nanoparticles. The diffraction pattern exhibits prominent peaks located at 2θ angles of 29.4º, 48.7º, and 59.7º, which correspond to the (112), (200), and (312) lattice planes, respectively. These peaks confirm the crystalline nature of the synthesized CuFeS2 nanoparticles, with the observed pattern closely matching the characteristic peaks of tetragonal chalcopyrite CuFeS2 as documented in the Joint Committee on Powder Diffraction Standards (JCPDS) card no. 37-0471. Notably, the peak at 29.4º corresponding to the (112) lattice plane exhibits the highest intensity, indicating its predominant presence in the synthesized $CuFeS_2$ nanoparticles. The Scherrer formula [14] was applied to estimate the crystallite size, revealing an average particle size of approximately 14 nm. This result underscores the influence of the hydrothermal method in enhancing the crystalline structure and controlling the particle size of $CuFeS_2$ nanoparticles. Based on the structural parameters obtained from XRD analysis (Table 1), the synthesized $CuFeS_2$ nanoparticles exhibit distinct features indicative of their crystalline nature and internal strain. The full width at half maximum (FWHM) values corresponding to the (112), (220), and

(312) lattice planes are 0.44328, 0.68822, and 1.03884 radians, respectively. These FWHM values reflect the degree of peak broadening, providing insights into the $CuFeS_2$ crystallite size distribution and internal strain within the nanoparticles.

Furthermore, the dislocation density (δ) and microstrain (ε) parameters offer valuable information about the structural defects and strain- induced distortions present in the synthesized $CuFeS_2$ nanoparticles. The observed microstrain values provide insights into the extent of lattice distortion and internal stress within the $CuFeS_2$ nanoparticles. Overall, the structural parameters obtained from XRD analysis elucidate the crystalline characteristics, internal strain, and defect density of the synthesized $CuFeS_2$ nanoparticles, contributing to a comprehensive understanding of their structural properties and potential applications.

The Raman spectrum of the synthesized $CuFeS_2$ sample is depicted in Figure 2(B), revealing several distinct peaks within the 100-700 cm^{-1} range. Specifically, peaks are observed at wavenumbers of 151, 184, 323, 449, and 659 cm^{-1}. The presence of Cu-S bonds is indicated by peaks observed at 184 and 659 cm^{-1}, while the presence of Fe-S bonds is evidenced by the peak at 323 cm^{-1}. Additionally, a prominent peak corresponding to sulfur is observed at 183 cm^{-1}. The peak at 151 cm^{-1} suggests the presence of S-S bonds. The dominant signal at 659 cm^{-1} further confirms the presence of $CuFeS_2$ on the mineral surface of chalcopyrite. Chalcopyrite exhibits a complex stratified arrangement, with individual sulfur atoms within layers and sulfur dimers covalently bonded between layers. The observed Raman peaks provide insights into the molecular structure and bonding configuration of $CuFeS_2$, highlighting its unique characteristics and potential application [15-16].

Figure 2(C) presents the field emission scanning electron microscopy (FESEM) image of the synthesized $CuFeS_2$ sample. The FESEM imaging revealed an amorphous morphology, indicating the absence of well-defined crystalline structures. One possible reason for observing an amorphous morphology in synthesized $CuFeS_2$ nanoparticles via the hydrothermal method could be the rapid nucleation and growth kinetics under the specific reaction conditions employed. In the hydrothermal synthesis process, precursor materials are reacted in an aqueous solution under elevated temperature and pressure conditions, promoting the formation of nanoparticles through nucleation and subsequent growth [17]. Additionally, energy-dispersive X-ray spectroscopy (EDX) analysis was conducted to verify the elemental composition of the synthesized $CuFeS_2$ nanoparticles. Figure 2(D) shows the EDX spectrum, confirming the presence of copper (Cu), iron (Fe), and sulfur (S) elements in the sample. Importantly, no significant impurities were detected in the EDX analysis, underscoring the purity of the synthesized $CuFeS_2$ nanoparticles.

The UV-Visible absorbance spectra of the synthesized $CuFeS_2$, as depicted in Figure 3(A) within the wavelength range of 300-900 nm, revealed a distinct peak absorption observed prominently near 550 nm. This absorption behavior indicates the $CuFeS_2$ interaction with electromagnetic radiation in the visible region, suggestive of its potential optical applications. Further analysis involved the calculation of the $CuFeS_2$ band gap, achieved through the Tauc plot method illustrated in Figure 3(B), resulting in a determined value of 2.35 eV. The enhanced band gap observed in $CuFeS_2$ synthesized via the hydrothermal method can be attributed to several factors. The controlled growth conditions inherent to the hydrothermal synthesis process enable precise manipulation of reaction parameters, leading to the formation of materials with tailored electronic properties [18]. Additionally, this method facilitates the minimization of defects within the crystal lattice, ensures uniform particle size and distribution, and allows for better control over stoichiometry. Collectively, these factors contribute to the modification of the $CuFeS_2$ electronic band structure, ultimately resulting in the observed increase in the band gap, thus highlighting the efficacy of the hydrothermal method in tailoring the optical properties of $CuFeS_2$ for various applications

The surface area analysis of the synthesized $CuFeS_2$ nanoparticles, as depicted in Figure 3(C), involved examining the adsorption-desorption isotherms of nitrogen molecules to characterize the material's surface area and pore structure. The analysis revealed a significant surface area of 62 m^2/g and a corresponding pore size of 10 nm, as illustrated in Figure 3(D). Notably, the nitrogen adsorption-desorption isotherm displayed type III behavior with hysteresis loops, indicating the presence of mesoporous characteristics within the $CuFeS_2$ structure. The improved surface area observed for $CuFeS_2$ synthesized via the hydrothermal method can be attributed to several factors inherent to this synthesis technique [19]. Hydrothermal synthesis provides a controlled environment conducive to the formation of well-defined crystalline structures with high surface area-to-volume ratios. Additionally, the precise control over reaction parameters such as temperature, pressure, and pH facilitates the creation of nanoparticles with uniform size and distribution, thereby enhancing the overall surface area. Furthermore, the mesoporous characteristics observed in the synthesized CuFeS2 nanoparticles suggest the presence of interconnected pores, which can contribute to the increased surface area. Overall, the hydrothermal method enables the synthesis of $CuFeS_2$ nanoparticles with improved surface area, making them promising candidates for various applications. Moreover, hydrothermal synthesis allows for precise control over reaction parameters, leading to the formation of nanoparticles with uniform size and distribution. Furthermore, the hydrothermal environment promotes the growth of crystalline structures with high surface area-to-volume ratios. These factors collectively contribute to the enhanced surface area of $CuFeS_2$ nanoparticles synthesized via the hydrothermal method, rendering them promising candidates for various applications, including catalysis, sensing, and energy [20].

IV. CONCLUSION

In conclusion, the hydrothermally synthesized $CuFeS_2$ nanoparticles were characterized using XRD, Raman, FESEM, EDX, UV-Visible, and BET surface area analysis. XRD revealed the $CuFeS_2$ nanoparticles exhibits the tetragonal chalcopyrite $CuFeS_2$ structure and the average particle size was 14 nm, indicating that the hydrothermal process controls particle size. FESEM imaging showed an amorphous morphology of $CuFeS_2$, probably owing to fast nucleation and growth kinetics. Raman spectroscopy revealed its molecular structure and bonding arrangement. EDX examination showed $CuFeS_2$ nanoparticles elemental composition without contaminants. UV-Visible spectroscopy showed strong absorption and a band gap of 2.35 eV, increased by controlled growth conditions and hydrothermal defect reduction. Additionally, BET surface area study showed a mesoporous surface area of 62 m^2/g due to regulated synthesis and linked pore development. These results show that the hydrothermal approach can tune $CuFeS_2$ nanoparticles structural and optical characteristics, making them suitable for catalysis, sensing, and energy conversion.

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Figure Caption

Figure 1 Schematic representation for synthesis of $CuFeS_2$ nanoparticles
Figure 2 Synthesized $CuFeS_2$ nanoparticles (A) XRD Analysis (B) Raman spectrum (C) FESEMimages (D) EDX spectrum
Figure 3 Synthesized $CuFeS^2$ nanoparticles (A) UV-Visible absorbance spectrum (B) Tauc Plot (C) N_2 adsorption/ desorption (D) Pore size

Table 1 Calculated structural parameters of the prepared $CuFeS_2$ nanoparticles

ples	2θ	(hkl)	D spacing	WHM Radian)	Lattice constants		crystallite size (nm)	Average crystallite size (nm)	Dislocation Density(δ)	Microstrain (ε)
					Lattice a = b (Å)	Para meter c (Å)				
$CuFeS_2$	29.4	112	3.0390	0.44328	5.289	10.42	19.3	14	0.000196	0.001882
	48.7	220	1.8700	0.68822			13.2		0.000363	0.001694
	57.9	312	1.5926	1.03884			9.1		0.000577	0.002092

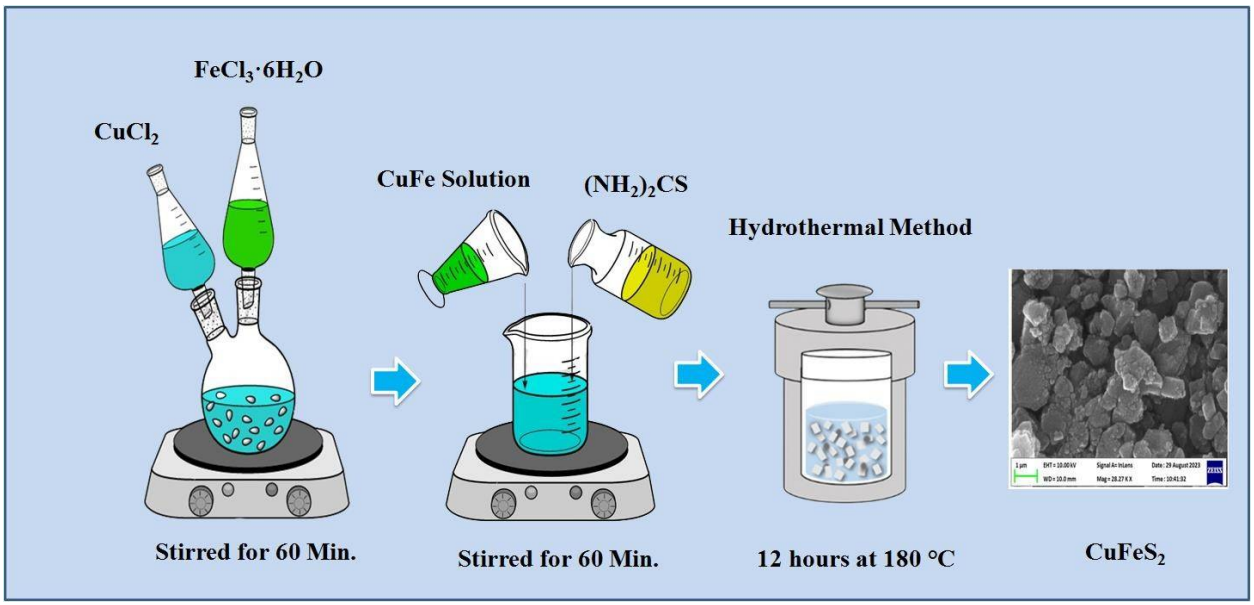


Figure 1 Schematic representation for synthesis of $CuFeS_2$ nanoparticles

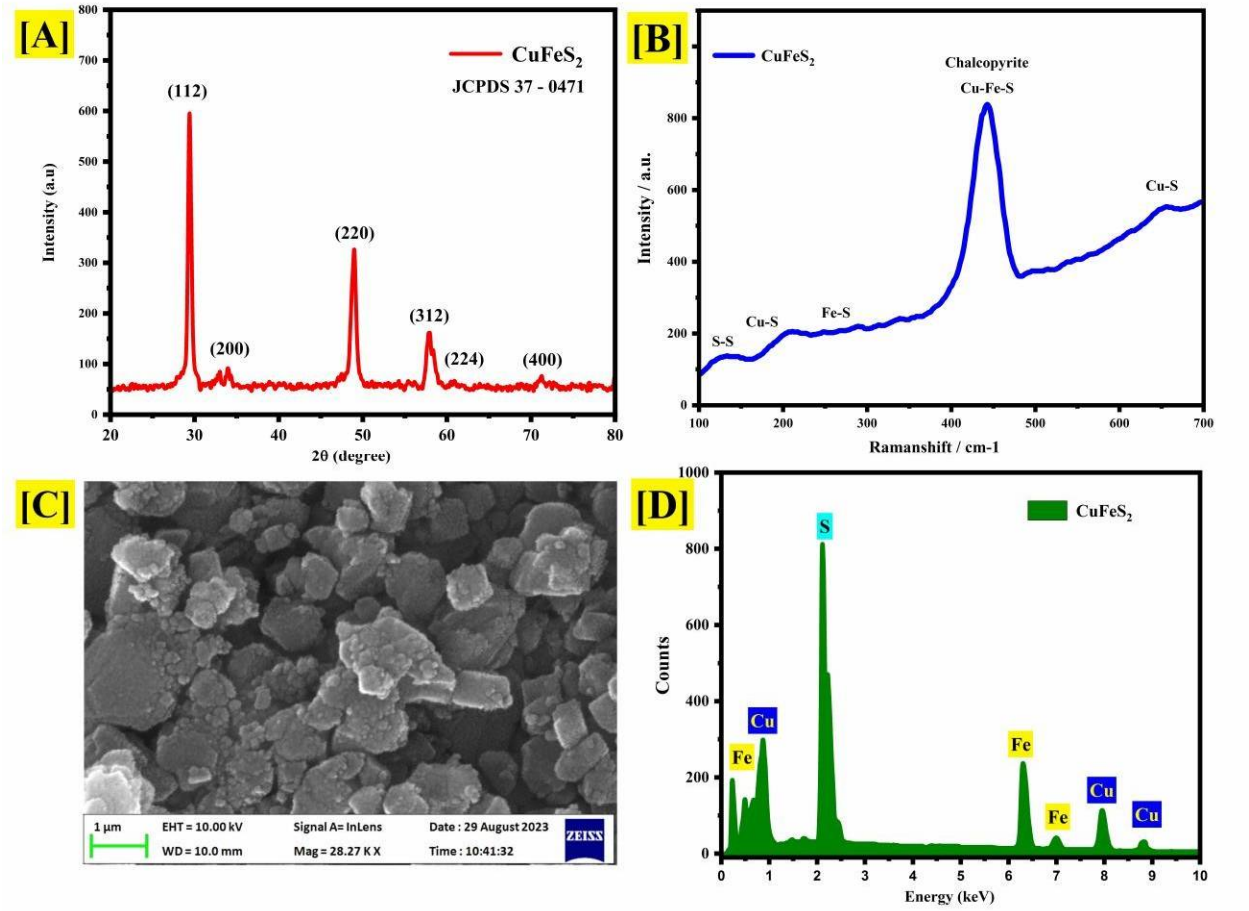


Figure 2 Synthesized $CuFeS_2$ nanoparticles (A) XRD Analysis (B) Raman spectrum (C) FESEM images (D) EDX spectrum

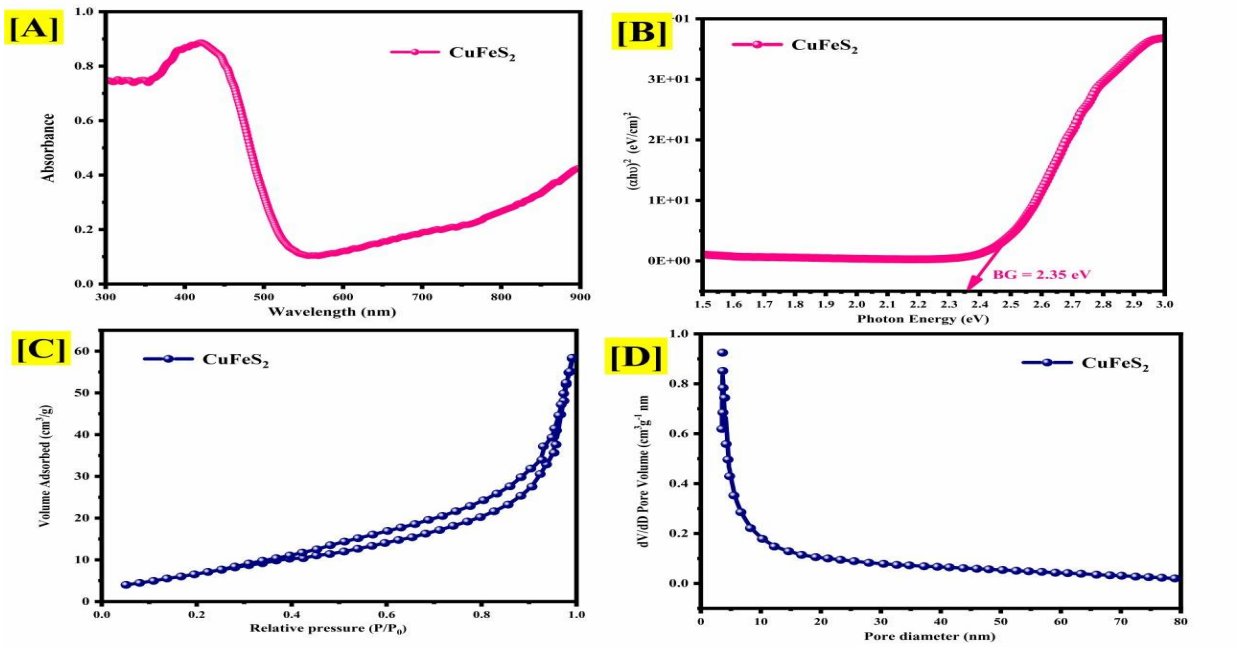
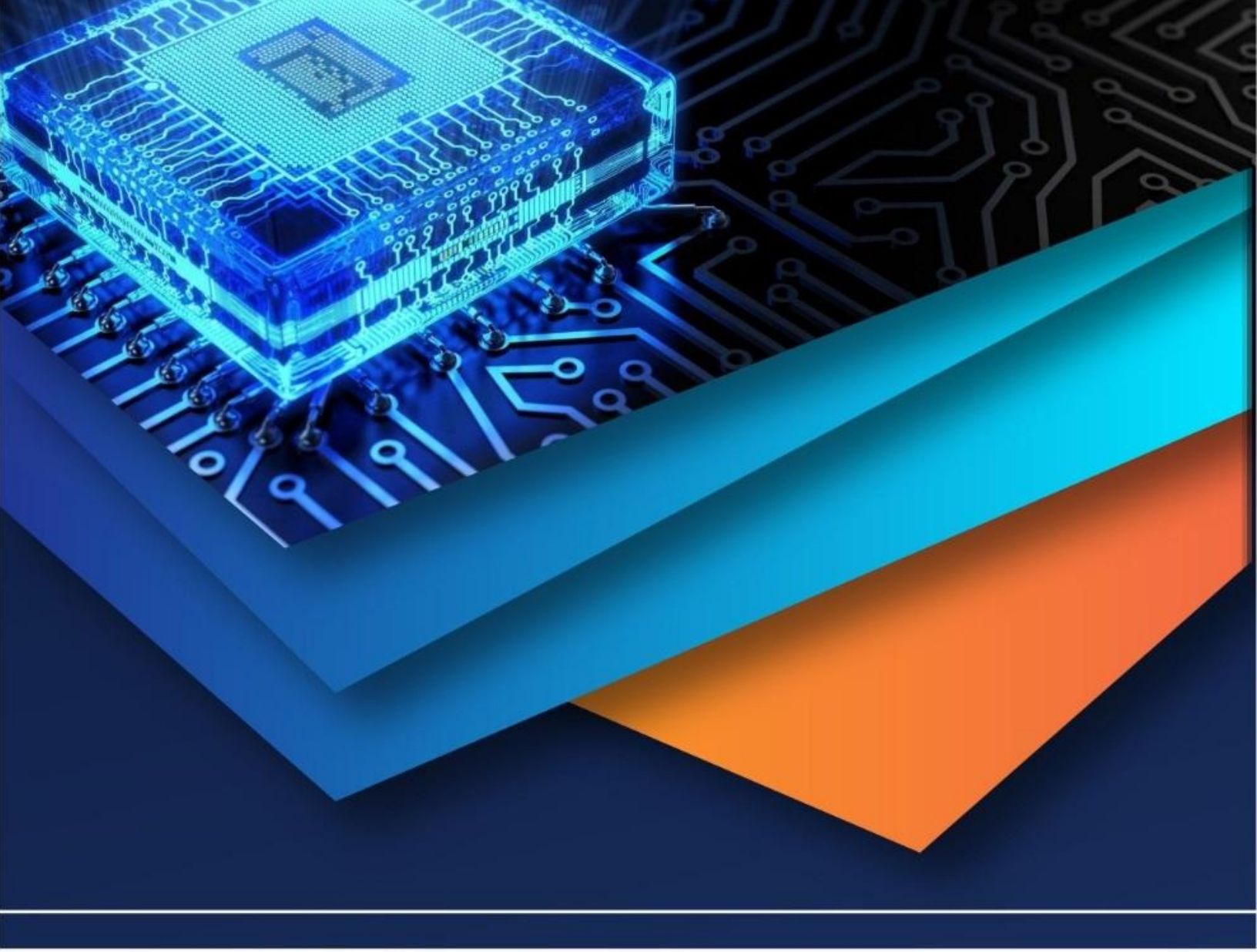


Figure 3 Synthesized $CuFeS_2$ nanoparticles (A) UV-Visible absorbance spectrum (B) Tauc Plot (C) N_2 adsorption/ desorption (D) Pore size



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