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Preparation and Characterization of CuFeS₂ Nanoparticles Synthesized via Hydrothermal Method

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Abstract: This research involved the successful synthesis of CuFeS₂ nanoparticles using a hydrothermal method. The CuFeS₂ 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 CuFeS₂ 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 BET surface area revealed a surface area of 62 m²/g and a pore size of 10 nm. The obtained findings suggest that the CuFeS₂ nanoparticles possess favorable properties that render them appropriate for use in photocatalytic applications.

Keywords: CuFeS₂, 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 (CuFeS₂) is a semiconductor compound that belongs to the I-III-VI₂ ternary group. The material has distinctive characteristics, including a high Neel temperature and outstanding electrical and optical capabilities, characterized by a very narrow optical band gap [6]. CuFeS₂ 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. CuFeS₂ nanoparticles possess these characteristics that render them very appealing for use in solar cells, photocatalysis, and photovoltaic systems [7].

Nevertheless, the achievement of successful synthesis and comprehensive characterization of CuFeS₂ 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 over their 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 CuFeS₂ 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 CuFeS₂ nanoparticles with tailored properties [9-10].

Understanding the structural and optical properties of CuFeS₂ nanoparticles is crucial for optimizing their synthesis and harnessing their potential in various applications. The crystal structure of CuFeS₂, 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; $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$; Sigma–Aldrich) were dissolved in 100 mL of pure water under continuous stirring for duration of 60 minutes. Following this, thiourea (1 mol, $(\text{NH}_2)_2\text{CS}$, 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) (XPRT-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, $\lambda = 200\text{--}900\text{ nm}$) 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 29.4° , 48.7° , and 59.7° , which correspond to the (112), (200), and (312) lattice planes, respectively. These peaks confirm the crystalline nature of the synthesized CuFeS_2 nanoparticles, with the observed pattern closely matching the characteristic peaks of tetragonal chalcopyrite CuFeS_2 as documented in the Joint Committee on Powder Diffraction Standards (JCPDS) card no. 37-0471. Notably, the peak at 29.4° 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 $100\text{--}700\text{ cm}^{-1}$ 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 $300\text{--}900\text{ nm}$, 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 $62\text{ m}^2/\text{g}$ 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 CuFeS_2 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 $62 \text{ m}^2/\text{g}$ 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 CuFeS₂ nanoparticles

Figure 2 Synthesized CuFeS₂ nanoparticles (A) XRD Analysis (B) Raman spectrum (C) FESEM images (D) EDX spectrum

Figure 3 Synthesized CuFeS₂ nanoparticles (A) UV-Visible absorbance spectrum (B) Tauc Plot (C) N₂ adsorption/ desorption (D) Pore size

Table 1 Calculated structural parameters of the prepared CuFeS₂ nanoparticles

Samples	2θ	(hkl)	D spacing	WHM (Radian)	Lattice constants		crystallite size (nm)	Average crystallite size (nm)	Dislocation Density (δ)	Microstrain (ε)
					Lattice a = b (Å)	Parameter 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

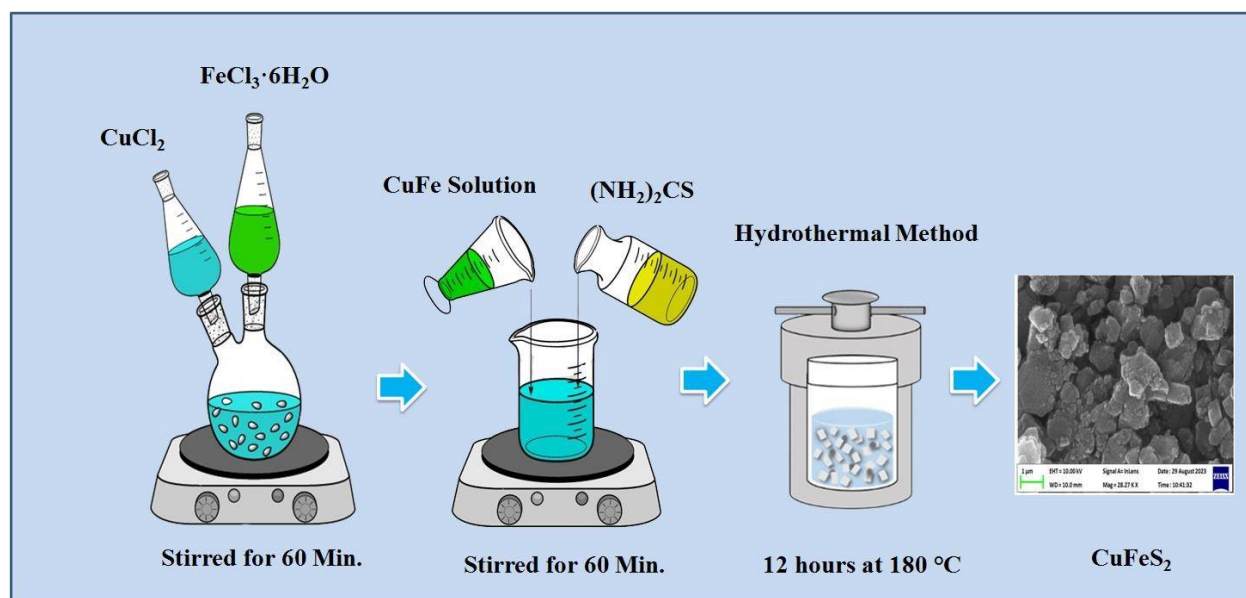


Figure 1 Schematic representation for synthesis of CuFeS₂ nanoparticles

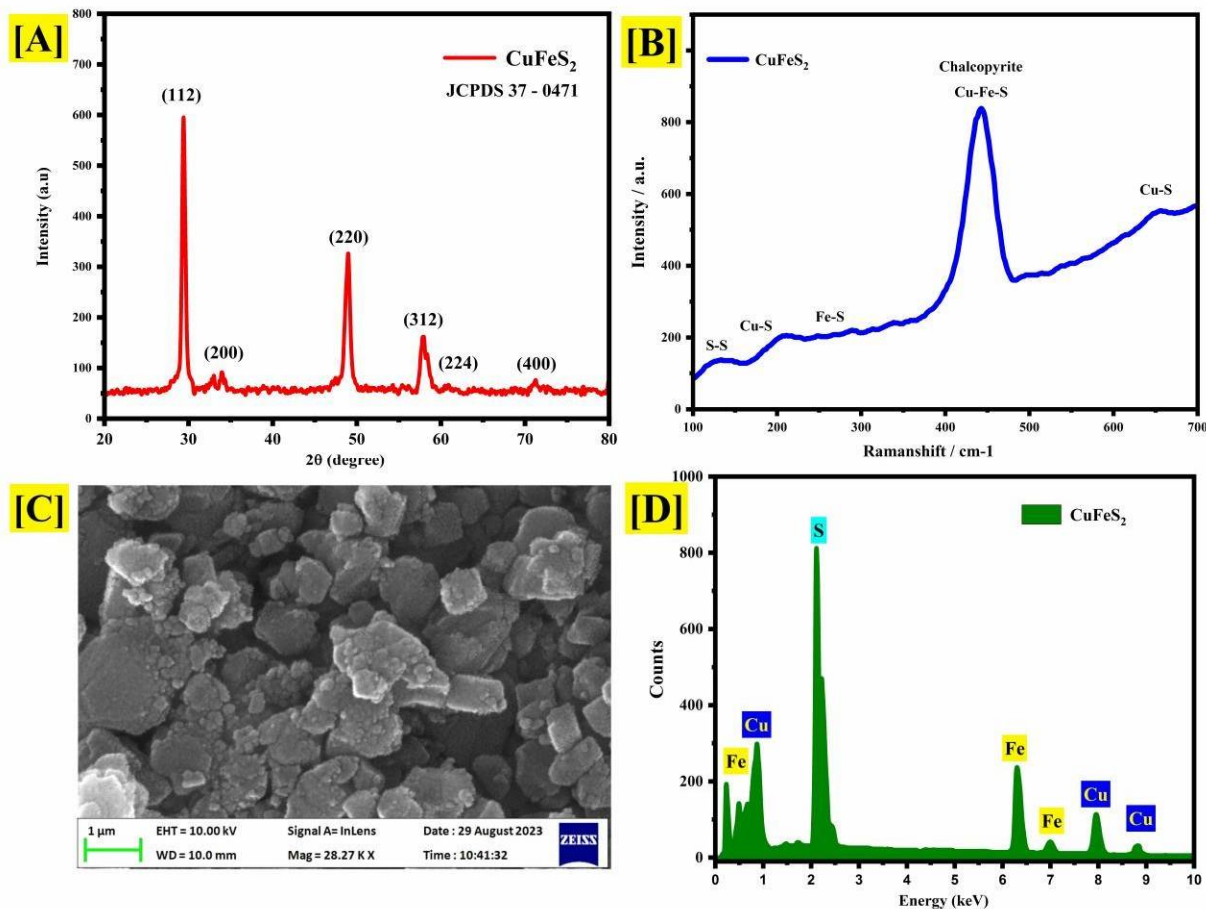


Figure 2 Synthesized CuFeS_2 nanoparticles (A) XRD Analysis (B) Raman spectrum (C) FESEM images (D) EDX spectrum

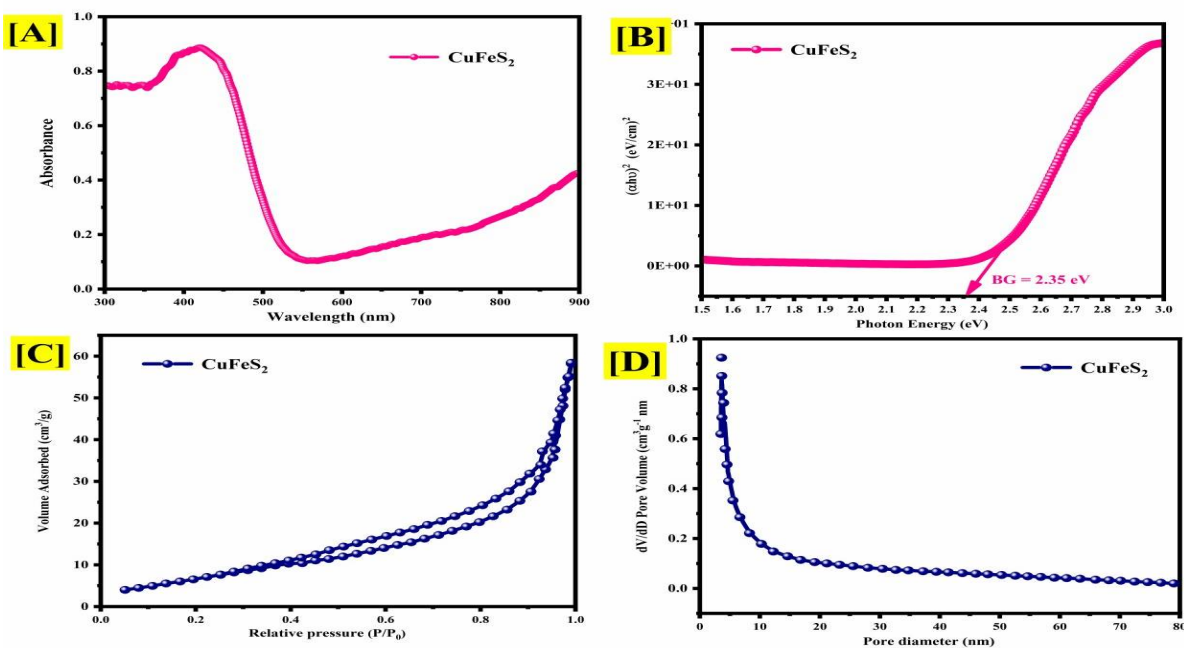


Figure 3 Synthesized CuFeS_2 nanoparticles (A) UV-Visible absorbance spectrum (B) Tauc Plot (C) N_2 adsorption/ desorption (D) Pore size



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