



One-step hydrothermal synthesis of CuS/MoS₂ composite for use as an electrochemical non-enzymatic glucose sensor

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ABSTRACT

Early diagnosis may be crucial for the prevention of chronic diabetes mellitus. For that herein, we prepared a CuS/MoS₂ composite for a non-enzymatic glucose sensor through a one-step hydrothermal method owing to the synergetic effect of CuS/MoS₂. The surface morphology of CuS/MoS₂ was studied by Field Emission Scanning Electron Microscopy (FESEM) and Cs-corrected Scanning Transmission Electron Microscopy (Cs-STEM). The crystallinity and surface composition of CuS/MoS₂ were analyzed by X-ray Diffraction (XRD) and X-ray Photoelectron Spectroscopy (XPS) respectively. The working electrode was prepared from CuS/MoS₂ electrocatalyst, and for that dispersed solution of electrocatalyst was used to fabricate the material-loaded glassy carbon electrode (GC). CuS/MoS₂ composite shows the viability of electrocatalyst to oxidize glucose in an alkaline solution with sensitivity and detection limit of 252.71 $\mu\text{A mM}^{-1} \text{cm}^{-2}$ and 1.52 μM respectively. The proposed glucose sensor showed reasonable stability and potential selectivity during electrochemical analysis. Accordingly, the CuS/MoS₂ composite has potential as a viable material for glucose sensing in diluted human serum.

1. Introduction

Diabetes is a chronic health condition resulting from glucose metabolism disorder in the human body [1–4]. “According to the World Health Organization (WHO), the number of deaths from diabetes increased globally by 70 % between 2000 and 2019” [5]. Prevention, diagnosis, and treatment require the development of fast, reliable, and accurate tools to detect blood glucose levels. Currently, a variety of analytical techniques including colorimetric and electrochemical are being used for glucose sensing [6,7]. Nowadays, electrochemical-based methods are attracting researchers’ attention due to their cost effectiveness, sensitivity, and stability. Based on enzyme use, electrochemical biosensors are classified into enzymatic and non-enzymatic biosensors. As reported by Wilson and Turner, enzymatic glucose sensors exhibit excellent selectivity and sensitivity, but poor stability at room temperature impairs their practical application [8,9]. So employing a catalyst like platinum [10], gold [11], silver [12], copper [13], as well as zinc

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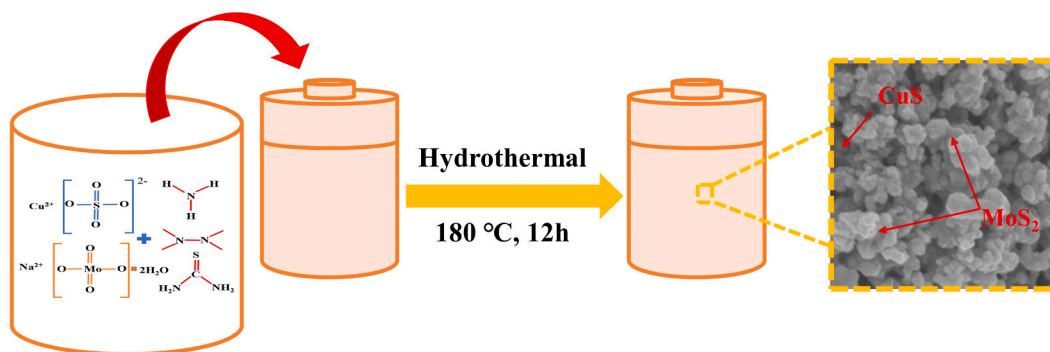


Fig. 1. Scheme showing hydrothermal process for CuS and CuS/MoS₂ synthesis.

metals or metal oxides/sulfide [14–16] instead of enzymes may be the one solution that helps to improve their stability and electrochemical properties. Among these, nanocomposites employing copper in different forms, such as Cu, CuO, Cu₂O, Cu(OH)₂, and CuS, exhibit interesting redox properties due to their Cu²⁺/Cu³⁺ redox pairs that contribute to electrochemical glucose oxidation [17–21]. However, electrochemical glucose oxidation is significantly dependent on the morphology and surface area. Binary copper sulfides occur in two structural forms, CuS (Covellite) and Cu₂S (Chalcocite) [22]. Copper chalcogenides are cost-effective materials that show tunable surface morphology with changes in parameters such as temperature, time, reactant, and reducing agent concentration [23, 24]. This morphology tuning results in excellent optical, electronic, electrochemical, and other physical properties that make the copper chalcogenides-based materials applicable to the Li-ion batteries [25], photocatalysts [26], solar cells [27], supercapacitors [28] and sensors [21]. Despite these benefits, low electron transfer, as well as poor electrical conductivity and dispersion, significantly diminish its effective catalytic activity [29]. To overcome this problem, researchers have prepared composite materials with a carbon-based component [3], including graphene [23], MXene [30], and molybdenum disulfide (MoS₂) [31]. The resulting materials feature increased conductivity as well as surface area of composite material, which play a vital role in the better electrocatalytic activity of electrocatalysts [32].

Molybdenum disulfide (MoS₂), a 2-D transition metal dichalcogenide with a typical band gap of 1.8 eV, exhibits a large surface area, low friction coefficient, better catalytic and physiochemical properties [33–35]. As a graphene analog, MoS₂ may be a viable electrode material for the electrochemical sensors. However, agglomeration, intrinsically low catalytic activity, and low conductivity have so far prevented its broad application [31,36]. So that researchers have attempted to overcome these problems by altering the surface morphology of MoS₂, combining it with supportive functional materials based on carbonaceous materials, conducting polymers, and metals/metal oxides/sulfides nanoparticles resulting in a unique structure and better catalytic properties in the composite material [31,37–39]. Based on this assumption, we used a one-step hydrothermal technique for the first time to prepare a bimetallic CuS/MoS₂ composite for a non-enzymatic electrochemical glucose sensor. The CuS/MoS₂ greatly enhanced the electrocatalytic properties improving ionic mobility, because of synergetic catalytic properties.

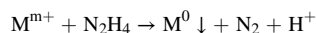
2. Materials and methods

The synthesis methods, chemicals used, characterization employed, electrode fabrication, and electrochemical measurement were explained in the supporting information (SI) of the manuscript. The preparation method involved is represented in Fig. 1.

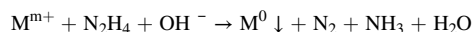
3. Results and discussion

The CuS/MoS₂ composite material synthesis procedure involves the ammonia solution, copper sulfate (copper source), sodium molybdate dihydrate (molybdenum source), thiourea (sulfur source), and hydrazine hydrate (reducing agent), where the hydrazine reduces different metal cations (M^{m+}) to the elemental state (M⁰), that undergoes self-oxidation/reduction reaction both in acidic and basic medium as represented in Fig. 1 scheme. Simultaneously, ammonia reacts with copper to form a copper ammonia complex from a complex formation reaction [40]. The reaction can be summarized as,

According to Lee, 1996. [41],

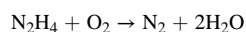


Based on Tobe and Burgess 1999. [42] discussion, the reduction reaction follows;



Similarly,

According to Audrieth and Ogg, 1951. [43], hydrazine reacts with dissolved oxygen in the water;



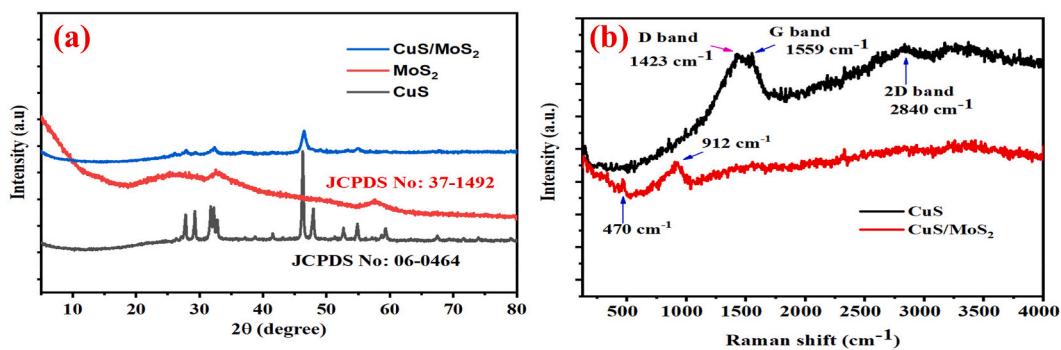


Fig. 2. XRD patterns (a), and Raman spectra (b) of CuS, and CuS/MoS₂.

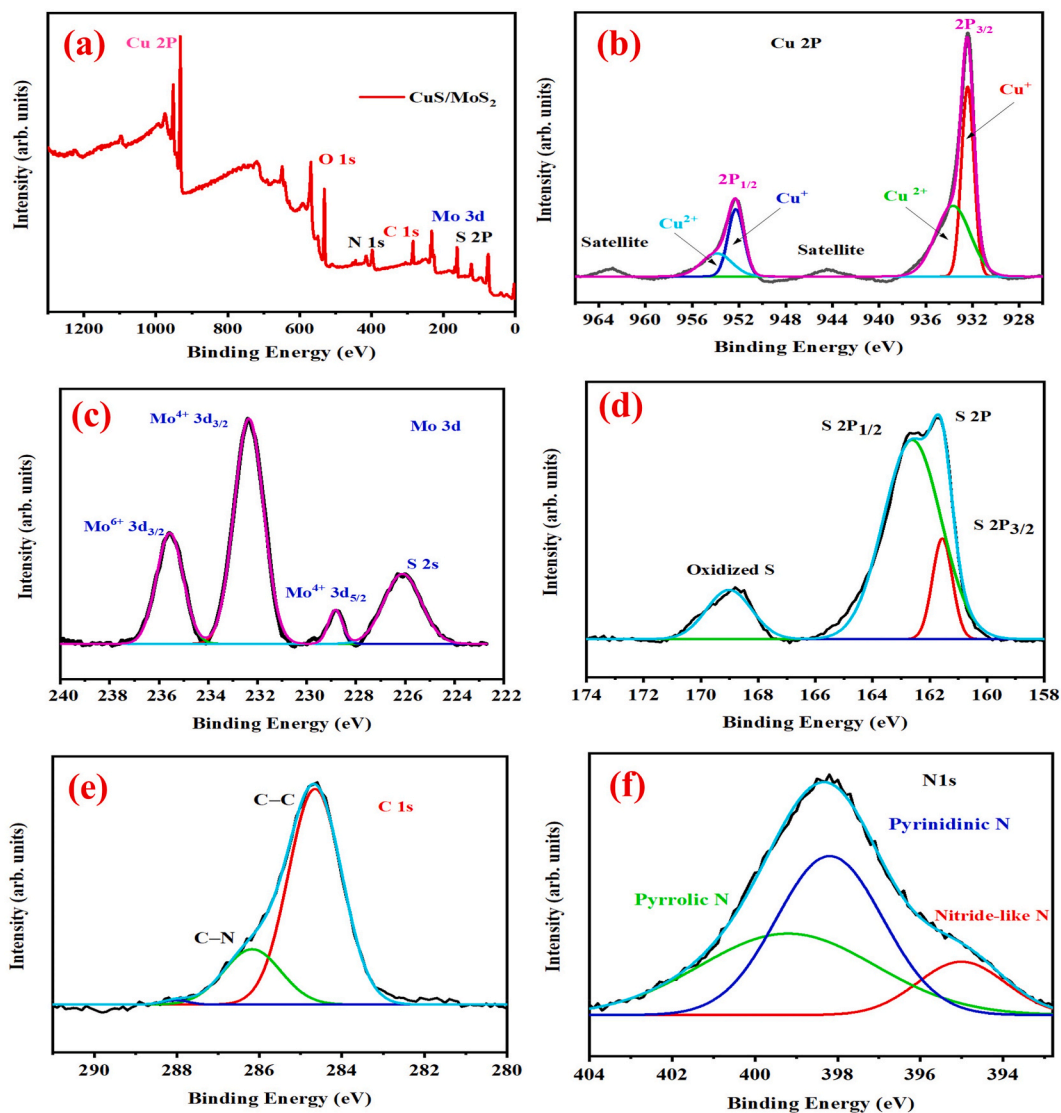


Fig. 3. (a) XPS survey spectrum, (b) Cu 2P, (c) Mo 3d, (d) S 2P, (e) C 1s, and (f) N1s high-resolution XPS spectra.

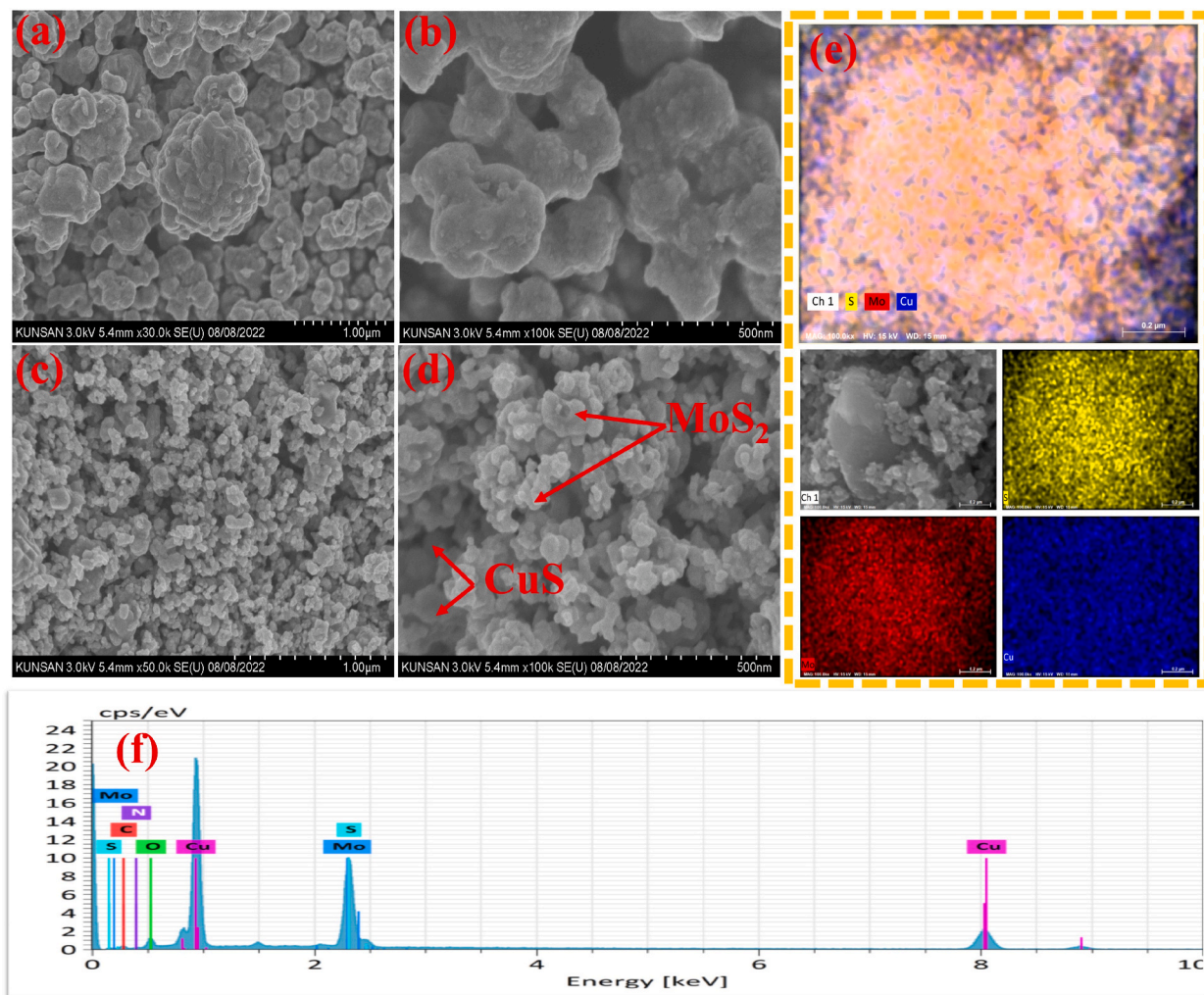
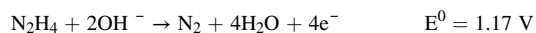
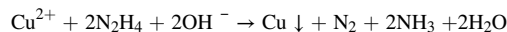
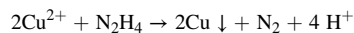


Fig. 4. (a, b) CuS, and (c, d) CuS/MoS₂ FE-SEM images, (e) corresponding EDX mapping, and (f) EDX spectrum of CuS/MoS₂.

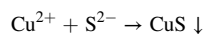
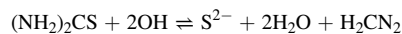
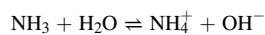
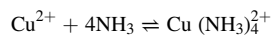
The half-reaction involved in the reduction reaction involving hydrazine is as follows [44];



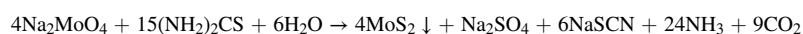
The reaction between the Cu²⁺ ion and hydrazine can be summarized as,



The possible mechanism for the copper sulfide assumes that the Cu²⁺ reacts with the S²⁻ ions produced by the hydrolysis of copper salt and thiourea which undergoes a sulfurization reaction during the hydrothermal reaction. The reaction mechanism of CuS and MoS₂ can be described as follows [45,46].



The reaction of sodium molybdate and thiourea in an aqueous medium can be summarized as,



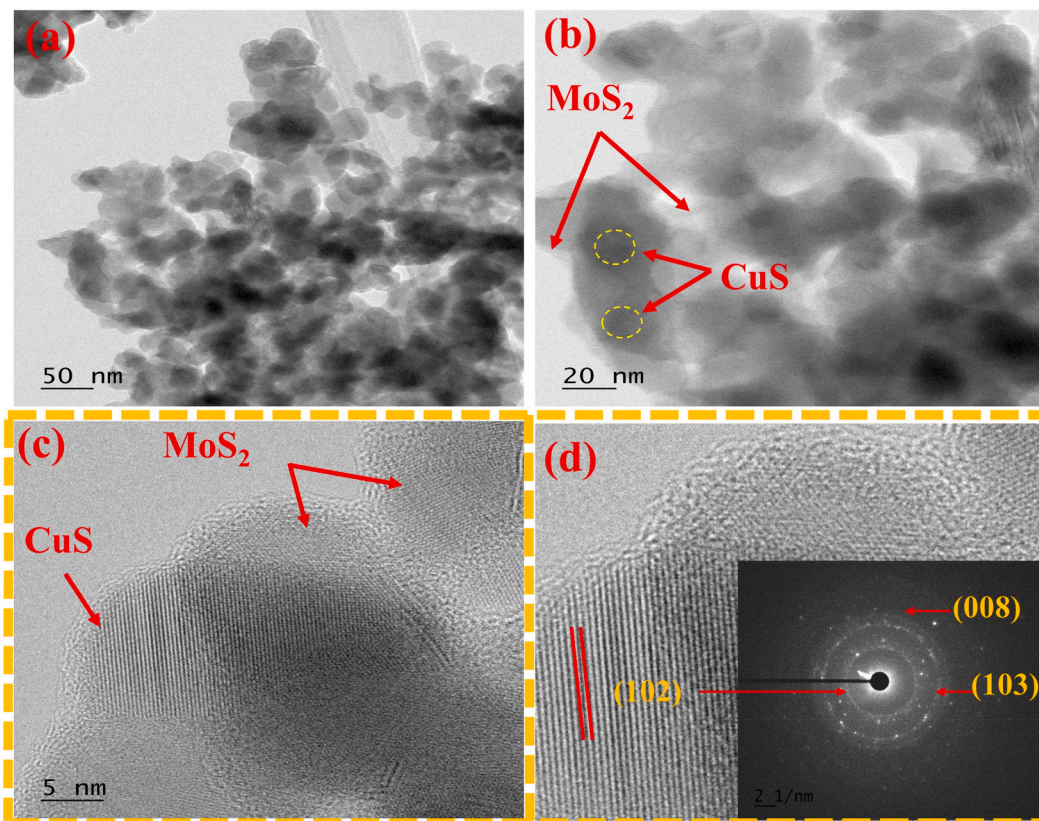


Fig. 5. TEM images (a, b), HR-TEM image (c), and magnified HR-TEM image with d-spacing and SEAD pattern (d) of CuS/MoS₂.

The crystalline phase structure of the composite was characterized by XRD, as illustrated in Fig. 2 (a). The XRD patterns with angle 2θ and crystal planes of the CuS were - 27.84° (101), 29.33° (102), 32.32° (103), 46.28° (008), 47.9° (110), 52.65° (108), 54.92° (104), 59.37° (203), 67.48° (203), 71.72° (207), 74.01° (208). This result resembles prior reported literature (JCPDS No. 06–0464) [3,47,48]. Furthermore, MoS₂ with angle 2θ and corresponding crystal planes: 32.71° (101) and 57.64° (004) shows quite close accordance with the reported JCPDS No. 37–1492 file [49–51]. The decrease in crystallinity of CuS/MoS₂ composite that possesses a combined peak of CuS and MoS₂ may be accounted for by the dispersion of the MoS₂ nanosheet over the CuS nanostructure, which may have occurred as a result of the combined activity of the hydrazine (reducing agent) and ammonia (complexing agent) [40,52,53]. Raman spectrum of CuS with peaks at 470 cm⁻¹ and 912 cm⁻¹ corresponds to the S–S stretching and C–N linkage between thiourea and ammonia, respectively, as indicated in Fig. 2(b) [53–59]. Similarly, the peaks indicating D band (1423 cm⁻¹), G band (1559 cm⁻¹), and 2D (2840 cm⁻¹) band highlights the presence of a carbon structure in the CuS [60–63] and the emergence of new bond confirms the creation of a bimetallic sulfide CuS/MoS₂ composite.

The XPS spectrum of the CuS/MoS₂ composite reveals the elemental composition and binding energies as represented in Fig. 3(a). XPS spectrum of CuS/MoS₂ reflects the atomic intensity of Cu, Mo, S, C, and N [3,64,65]. Fig. 3 (b) depicts the deconvolution peaks at 932.34 eV and 952.25 eV binding energies representing the Cu⁺ 2p_{3/2} and Cu⁺ 2p_{1/2}, respectively. Similarly, two more deconvolution peaks at 933.61 eV and 953.87 eV correspond to Cu²⁺ 2p_{3/2}, and Cu²⁺ 2p_{1/2} respectively. Furthermore, satellite peaks at 944.44 eV and 962.84 eV correspond to the paramagnetic chemical state of the Cu²⁺ [66–70]. As shown in Fig. 3 (c), the characteristics peaks at 235.5 eV, 232.3 eV, 288.8 eV, and 226.1 eV indicate the predominance of, Mo⁶⁺ 3d_{3/2}, Mo⁴⁺ 3d_{3/2}, Mo⁴⁺ 3d_{5/2}, and S 2s, respectively [31,71–73]. The peaks represented in Fig. 3 (d), specify deconvolution peaks situated at 161.5 eV, 162.5 eV, and 168.9 eV indicating S 2p_{3/2}, S 2p_{1/2}, and oxidized S respectively [3,74–77]. Fig. 3 (e) represents fitted peaks at 284.6 eV and 286.1 eV assigned to C–C and C–N, respectively [3,65,78,79]. As represented in Fig. 3 (f), peaks at 394.9 eV, 298.1 eV, and 299.3 eV, correspond to nitride-like-N, pyridinic-N, and pyrrolic-N, respectively [65,80–83].

Surface morphology was characterized by using FE-SEM (in Fig. 4), and HRTEM (Fig. 5) [84]. The FE-SEM images of CuS at 30 K and 100 K magnification revealed its aggregated ball-like nanostructures (Fig. 4 (a, b)) [85]. Furthermore, the FE-SEM image of CuS/MoS₂ indicates the MoS₂ nanosheet grown at the CuS surface (Fig. 4 (c, d)) [85]. The surface morphology modification after synergy between CuS and 2-D MoS₂ decreases agglomeration and increases sufficient electrochemically active sites by increasing surface area resulting in better electrocatalytic activity [86,87]. As shown in Fig. 4. (d), the FE-SEM image of the CuS/MoS₂ composite reflects a clear surface morphology with two different components (CuS and MoS₂). The EDX color mapping of CuS/MoS₂ composite (Fig. 4. (e)) and EDX spectrum of CuS/MoS₂ composite (Fig. 4. (f)) indicates the presence of CuS and MoS₂ in the CuS/MoS₂ composite

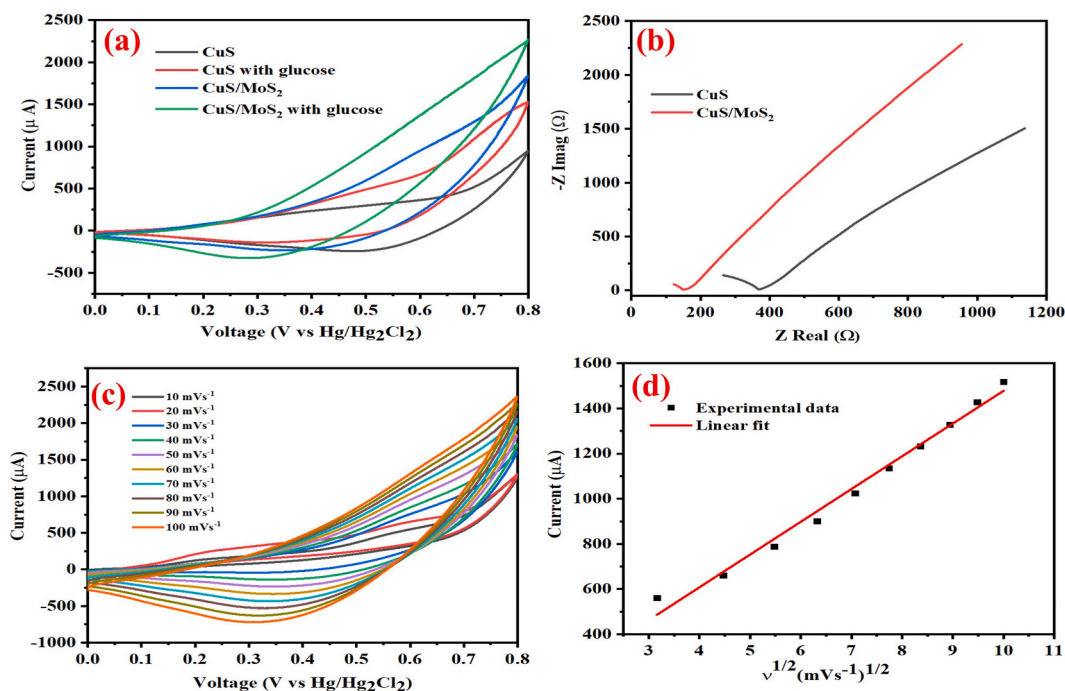


Fig. 6. CV spectra with and without glucose (a), Nyquist plot of CuS and CuS/MoS₂ (b), CV of CuS/MoS₂ at various scan rates (c), linear fit of sq. root of scan rate against oxidation current (d).

material [3,88].

The morphology of the CuS/MoS₂ was analyzed using Cs-STEM micrographs. The result revealed that the CuS was wrapped with dispersed molybdenum nanosheets (Fig. 5. (a, b)). The HR-TEM image of CuS revealed 0.32 nm lattice spacing with crystal plane (102) (Fig. 5 (c and d)). The SEAD pattern of CuS/MoS₂ composite indicates polycrystallinity with circular rings that support the crystal planes (102), and (103) and the (008) of CuS (inset image in Fig. 5(d).) [89–91].

3.1. Electrochemical glucose sensing

The electrocatalytic activity of CuS/MoS₂ and CuS were analyzed without and with glucose addition (2 mM) using 0.1 M NaOH electrolyte in a three-electrode system. Herein, we can figure out the gradual current increment in CuS/MoS₂ electrode as compared to the CuS at 50 mVs⁻¹ in cyclic voltammetric response shows the improved electrocatalytic activity of the CuS/MoS₂ composite. Similarly, in CuS and CuS/MoS₂ electrode oxidation current increment after the addition of glucose signifies the feasibility of electrode material towards non-enzymatic glucose sensor, as shown in Fig. 6 (a) [3,65,92]. The increased current response is due to the synergy of bimetallic sulfides and increased dispersion due to molybdenum disulfide nanosheets [93]. The HR-TEM image of the CuS/MoS₂ nanostructure indicates the accessible sites that promote electron transfer during the electro-oxidation of glucose [94]. The morphological structure of the CuS/MoS₂ composite suggests that the dispersion of the MoS₂ nanosheet over the CuS nanostructure, which increases potential active site and improves electrolytic dispersion, in turn, enhances electrochemical glucose oxidation through Cu (II/III) mediated electrochemical reversible redox reactions in an alkaline electrolyte, which are as summarized below [3,65,95, 96].



We performed an EIS analysis of CuS/MoS₂ and CuS electrodes in an equal volume ratio of ferricyanide solution (5 mM) and potassium chloride (0.1 M) solution at 0.1 Hz to 100 kHz frequency as shown in Fig. 6(b). Slope increment of the CuS/MoS₂ in the higher frequency region as compared to CuS suggests better electrocatalytic property of CuS/MoS₂ [85,97], that corresponds to synergetic behavior of the CuS and MoS₂ in CuS/MoS₂ composite [3].

Fig. 6(c) shows a cyclic voltammogram of CuS/MoS₂ composite with current response and scan rate relationship. The increase in anodic oxidation current along with concerning scan rates (10–100 mVs⁻¹) demonstrates the linear dependence [65]. Solving the regression equation obtained from linear regression between oxidation peak current response vs square root of scan rate we get $R^2 = 0.987$, which indicates the potentiality for the diffusion-controlled electrochemical reaction at the electrode surface of CuS/MoS₂

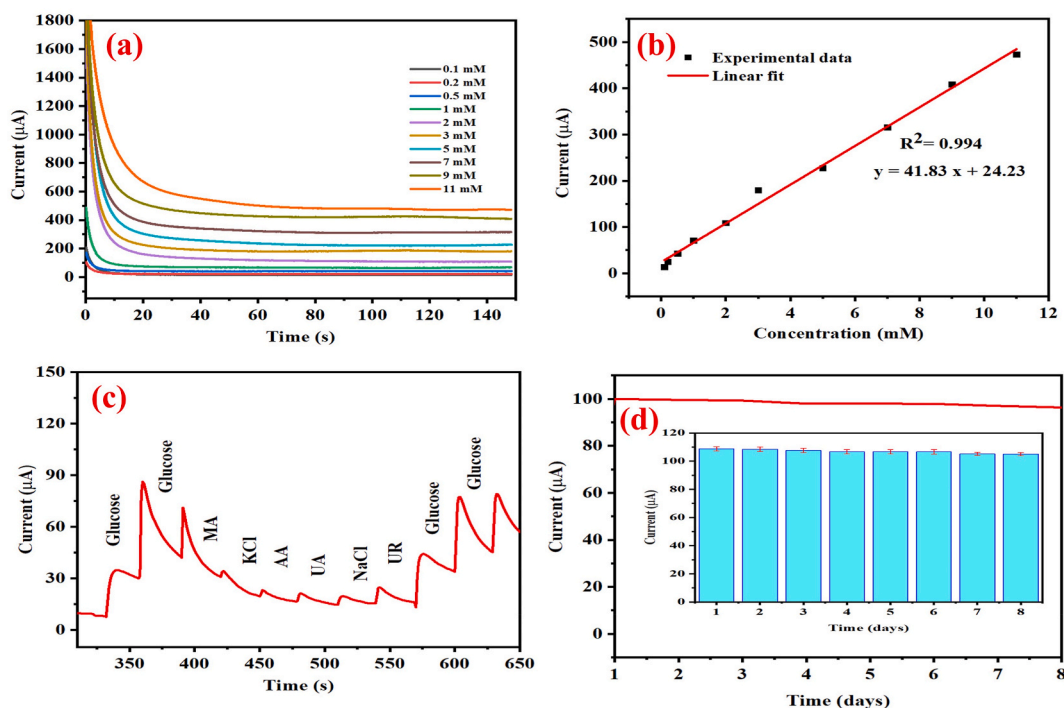


Fig. 7. Chronoamperometry curves of CuS/MoS₂ samples with glucose concentration (100 µM –11 mM) (a), linear fitting of Current Vs. concentration plot of CuS/MoS₂ (b), interference study using glucose and potential interfering species (c) and Stability test of CuS/MoS₂ electrode to 2 mM glucose showing change over time (days) (d).

Table 1

The comparison table of Cu- and Mo-based electrodes for glucose sensing.

Electrode material	Linear range	Detection limit	Sensitivity	Ref.
CuS MF	0.02–5.4 mM	2.0 µM	1007 µA mM ⁻¹ cm ⁻²	[21]
Bilayer MoS ₂	30–300 mM	300 nM	260.75 µA mM ⁻¹ cm ⁻²	[99]
MoS ₂ -CuNPs	4–4000 µM	–	1055 µA mM ⁻¹ cm ⁻²	[100]
MoS ₂ -CuS	1–100 µM	0.3 µM	–	[101]
CuO/MoS ₂	0.1–1 mM	0.017 µM	1055 µA mM ⁻¹ cm ⁻²	[94]
Cu _x S-MoS ₂ -rGO	2–720 µM	0.6 µM	308.2 µA mM ⁻¹ cm ⁻²	[31]
CuS nanotube	0.05–5 µM	–	7.842 µA mM ⁻¹ cm ⁻²	[102]
Cu-Cu ₂ S	0.002–8.1 mM	0.1 µM	361.58 µA mM ⁻¹ cm ⁻²	[103]
CuS/MoS ₂	0.1–11 mM	1.52 µM	252.71 µA mM ⁻¹ cm ⁻²	This work

composite. The electron transfer kinetics in electrochemical reaction occurs as a result of charge separation through a diffusion-controlled reaction mediated by charge separation and diffusion process due to MoS₂ nanosheet, which is shown in Fig. 6(d) [65,85,94].

3.2. Chronoamperometric analysis

The chronoamperometric spectra of CuS/MoS₂ are represented in Fig. 7(a) [3,85]. The result depicts that the gradual addition of glucose in the concentration range (100 µM- 11 mM) into an alkaline medium solution gives rise to the current increment. This result supports the idea that this material may have a practical application as an electrode material capable of monitoring glucose levels at 0.5 V applied potential. On plotting the oxidation current against the concentration, we get a linear fitting with R² value of 0.994, as shown in Fig. 7(b) [3]. The sensitivity of the CuS/MoS₂ electrode-based glucose sensor was estimated using $S = m/A$, in which m and A respectively represent the slope obtained from the calibration plot and the electrochemical active surface area ($A = 0.1655 \text{ cm}^2$) of the electrode. The limit of the detection LOD was calculated using the formula; $\text{LOD} = 3\sigma/m$, (where, σ = standard derivation, and m = slope) [3,85,94,95,98]. The sensitivity and detection limit were found to be 252.71 µA mM⁻¹cm⁻² and 1.52 µM, respectively. Table 1 compares linear range, detection limit and sensitivity of related materials developed and described by elsewhere.

Table 2
Determination of glucose in diluted human serum.

Sample	Added glucose concentration (μM)	Found glucose concentration (μM)	Recovery (%)	RSD (%)
1	500	483	96.60	1.29
2	1000	969	96.90	1.00
3	2000	1966	98.30	1.10

3.3. Selectivity and stability analysis

To prove the better selectivity a sensor should be able to detect interfering reagent species along with glucose during electrochemical reaction in chronoamperometry analysis. For that, the selectivity of CuS/MoS₂ electrode-based glucose sensor was checked by subjecting glucose solution (1.0 mM) as well as interfering reagent solutions such as ascorbic acid (AA), maleic acid (MA), potassium chloride (KCl), urea (UR), uric acid (UA), and sodium chloride (NaCl), (0.5 mM) [3,70]. The simultaneous oxidation of interfering agents and glucose during an electrochemical reaction demonstrates the selectivity of the CuS/MoS₂ as shown in Fig. 7(c) [3,85]. The selectivity of the CuS/MoS₂ electrode over potential interfering reagents is proved by the relatively low current response as compared to the glucose in an electrochemical reaction [3]. The stability was estimated by chronoamperometric tests over eight days. The sensitivity of CuS/MoS₂ was found to have decreased by 3.26 % over that time, showing 96.47 % stability (Fig. 7(d)). This reproducibility is thus quite adequate for an electrode material.

3.4. Real sample analysis

For the real sample analysis, a standard addition method was used to measure the recovery percentage by putting glucose solution of known concentration into diluted normal human serum (H4522, Sigma-Aldrich, 4.89 mM) [3] sample in alkaline medium. As tabulated in Table 2 the CuS/MoS₂ based electrode demonstrated a recovery percentage of 96.60 %, 96.90 % and 98.30 %, in response for the 500 μM , 1000 μM , and 2000 μM concentration of glucose solution [85]. These results suggest that the CuS/MoS₂ based electrode is viable for non-enzymatic glucose sensor as proven by a relative standard deviation (RSD) of less than 1.3 % [3,65,104].

4. Conclusions

We synthesized CuS/MoS₂ through one-step hydrothermal method. The structural morphology of the nanocomposite was characterized using diverse techniques to assess its suitability for the electrochemical non-enzymatic glucose sensor. The morphological structure of the CuS/MoS₂ provides an active site for glucose electrooxidation. The composite exhibits a sensitivity of 252.71 $\mu\text{A mM}^{-1}\text{cm}^{-2}$, a linear range 100 μM -11 mM, and a detection limit of 1.52 μM . The CuS/MoS₂-based glucose sensor's remarkable selectivity towards interfering species, as well as its stability, suggest that it has potential as a non-enzymatic electrochemical glucose sensor.

Data availability statement

Data will be made available on request.

CRediT authorship contribution statement

Krishna Prasad Sharma: Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Miyeon Shin:** Writing – review & editing, Software, Resources, Project administration, Data curation. **Ganesh Prasad Awasthi:** Software, Project administration, Methodology, Funding acquisition. **Soonhwan Cho:** Visualization, Software, Resources, Project administration, Methodology, Funding acquisition. **Changho Yu:** Writing – review & editing, Supervision, Project administration, Investigation, Funding acquisition, Formal analysis.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.heliyon.2023.e23721>.

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