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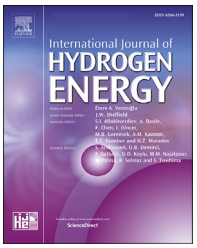

Synthesizing of SnS_2 photocatalyst from SnO_2 powders by thermal sulfurization with varying temperature (400 °C and 500 °C) and time

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HIGHLIGHTS

- SnS_2 succesfully synthesized from SnO_2 powders by thermal sulfurization method.
- Hexagonal SnS_2 powders obtained after optimization of sulfurization temperature and time.
- SnS_2 photcatlysis exhibited 2.5 μA/cm^2 photo current greater than literature values.

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ABSTRACT

SnS_2 from SnO_2 powders was successfully produced by the thermal sulfurization method to obtain pure SnS_2 powders by varying the sulfurization temperature and time. XRD analysis confirmed pure hexagonal SnS_2 powders at 400 and 500 °C after 24 h of sulfurization. Grain size analysis in SEM indicated that the average grain size of powders synthesized at 400 °C and 500 °C were 1 and 11 μm, respectively. The band gap values of the obtained powders at 400 and 500 °C was determined by UV–vis spectroscopy as 2.26 eV and 2.24 eV, respectively. The photoelectrochemical analysis of the SnS_2 photoelectrode produced at 500 °C revealed a flat band potential of −0.50 V, a charge carrier density of 1.49 × 10^{20} cm $^{-3}$ and photocurrent density of 2.5 μA/cm^2 at 0 V vs SCE. These results indicated that SnS_2 synthesized for the first time by the thermal sulfurization technique from SnO_2, which was a simple and cheap technique, was a promising candidate to be used as a photocatalysts.

1. Introduction

Hydrogen can be a solution to the climate crisis, as stated by the latest decision taken by the European Parliament and the Council on June 30, 2021. Hydrogen is one of the leading sources of renewable energy as an alternative to fossil fuels. Hydrogen is produced from both renewable and nonrenewable sources of energy. However, using non-renewable energy sources in hydrogen production will cause greenhouse gas emissions and global warming. The production of H_2 by splitting water with solar energy as a renewable energy source through photocatalytic and photo-electrochemical methods

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has great potential to be an alternative energy source to satisfy the world's energy needs [1–5].

Photocatalysts are semiconductor materials that are activated by light energy and initiate a series of electrochemical reactions. Many photocatalytic semiconductor materials have been studied, such as metal oxides, metal nitrides, oxynitrides, metal sulfides, and organic materials. Metal oxides (e.g., TiO_2) have suitable band structures that provide the thermodynamic requirements for splitting water, and they are resistant to photo-corrosion.

However, they are active under ultraviolet light, which constitutes 4% of the solar spectrum due to its wide band gap value, and because of this they have very low efficiency, which limits their uses [6,7]. On the other hand, metal sulfide (CdS, ZnS, CdZnS, SnS_2) [8–10] photocatalysts have suitable band gaps (2.2–3.2 eV) and valence and conduction band levels to harvest the visible light region of the solar spectrum. In this way, they have the advantage of possessing greater efficiency of H_2 by splitting water as compared to metal oxides (TiO_2). Due to their low oxidation ability, sacrificial electron donors (SO_3^{-2}) as an oxidation reaction for photocatalytic H_2 production were used [6,7]. Amongst metal sulfides, SnS_2 has an optimum band gap value of 2.2 eV. Because of this, it has a high potential to have a high H_2 production efficiency [11–13]. On the other hand, the rapid recombination of charge carriers, which lowers photocatalytic efficiency, is observed. Due to the enhanced recombination, only 10% of the excitons proceed through a photo-reduction incident [13]. Due to these desirable features mentioned above, studies on SnS_2 water splitting have increased rapidly in recent years, attracting the attention of researchers (Fig. 1). The number of publications related to "SnS_2 and water splitting" in the Scopus database since 2012 is given in Fig. 1, which demonstrates that there is still a long way to go in terms of splitting water using SnS_2 as a photocatalyst [13].

The literature commonly follows the hydrothermal method to produce SnS_2 in studies exploring its photocatalytic characteristics. Several studies have been conducted on this topic.

In the literature, there are a few studies examining the photocatalytic properties of SnS_2 synthesized by the thermal sulfurization method, a simple and affordable process.

Zhao et al. [14] synthesized 3D flower like SnS_2 nanoplates using a hydrothermal procedure, with the process taking a total of 37 h and beginning with $SnCl_4.5H_2O$ as the starting material. They calculated the band gap value of SnS_2 as 2.28 eV. In order to investigate the photoelectrochemical behavior they used an aqueous solution containing Na_2SO_4 as a sacrificial agent under 500 W Xe lamp irradiation with a 420 nm cutoff filter. The photocurrent density of SnS_2 was measured as 1.5 $\mu A/cm^2$.

Geng et al. [15] used a hydrothermal route starting from $SnCl_4.5H_2O$, with the procedure requiring 17 h for synthesizing SnS_2 which exhibited regular hexagonal nanosheet structures. After UV–vis diffuse reflectance spectra measurements, the band gap value of pristine SnS_2 was calculated as 1.99 eV. In photoelectrochemical experiments carried out in solutions containing Na_2SO_4, SnS_2 showed a 1.4 $\mu A/cm^2$ current density under 300 W Xe lamp illumination with a 420 nm cut-off filter.

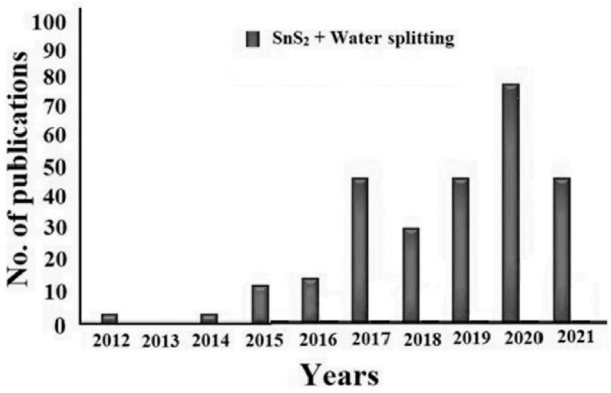

Fig. 1 – Number of publications found on Scopus using the keywords, "SnS_2 + Water Splitting" [13].

Liu Y. et al. [16]. synthesized hexagonal SnS_2 nanostructures using stannic chloride a thiourea as precursors in a hydrothermal method. They measured the indirect band gap of SnS_2 as 2.25 eV. In an aqueous solution containing 0.5 M Nas_2SO_4, the photocurrent response of SnS_2 photoelectrode was measured as 1.5 $\mu A/cm^2$ with a 0.5 V bias vs. Ag/AgCl electrode under a 300 W Xe lamp irradiation with a 400 nm cutting of filter.

There were a few studies in the literature where sulfurization process was employed to produce SnS_2 photocatalysis.

In their study, Huang [17] and colleagues used SnS thin films as precursors. They sulfurized SnS thin films at 250 °C for 2 h under a low vacuum with excess sulfur to produce hexagonal SnS_2 thin films. The obtained film's direct band gap value as 2.22 eV SnS_2 photoelectrodes showed 10–15 $\mu A/cm^2$ photocurrent density under 100 mW/cm^2 irradiation in a 0.25 M H_2SO_4 solution at 0 V vs Ag/AgCl.

Liu et al. [18] synthesized pure SnS_2 nanosheets from Sn nanoparticles by the sulfurization method using elemental sulfur as a sulfur source in sealed tube glass that was placed in a tube furnace. They set the sulfurization temperature at 300 °C and kept it there for 120 min. After that, they heat the obtained powder to 200 °C for 120 min in an alumina crucible to remove excess sulfur. Synthesized powders show hexagonal crystal structures. They measured the band gap value of SnS_2 powders as 2.15 eV. For photoelectrochemical measurements, they used a 300 W Xe lamp with a 420 nm filter for illumination, and they prepared a 0.5 M Na_2SO_4 solution as an electrolyte. In photocurrent transient response measurements, they reported that SnS_2 sample show 1 $\mu A/cm^2$ at a bias potential of 0.0 V vs. Ag/AgCl.

The study in this paper was aimed to fill this gap by examining the photocatalytic properties of SnS_2 synthesized from SnO_2 first time by a low-cost, one-step thermal sulfurization method. Also, it has been reported that in the author's previous work [8] photocatalysts synthesized by the thermal sulfurization technique displayed a distinct and exclusive hexagonal phase with high crystallinity, an absorption edge at the longest wavelength, and comparatively elevated photocatalytic activity in the absence of noble metal cocatalysts.

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Within the scope of this study, it was aimed to synthesize SnS_2 photocatalyst to produce H_2 with high efficiency by splitting water via solar energy. The sulfurization parameters, such as exposure temperature and time, were optimized for obtaining the highest purity of the SnS_2 phase and eliminating SnO_2. The photoelectrochemical characterization techniques (chronoamperometry, linear-sweep voltammograms, Mott-Schottky plots, and open circuit potential under dark and illuminated conditions) were used to determine the PEC properties of SnS_2 photocatalysis.

2. Materials and methods

2.1. Synthesis of tin disulfide (SnS2)

Tin disulfide (SnS_2) powders have been synthesized from tin oxide (SnO_2) nano powders (≤100 nm average particle size obtained from sigma Aldrich) by sulfurization using 99.9% pure sulfur powder in a Protherm hot-wall tube furnace at 400 and 500 °C to examine the effect of temperature on SnS_2 formation. Other phases (Sn_2S_3, SnS) are produced in sulfurization reactions conducted at 600 °C and above in addition to SnS_2 [20]. For these reasons, the temperature selected in the experiments did not exceed 500 °C. As indicated in Fig 2, 100 mg of SnO_2 powders were put in an alumina crucible and placed in the center of the heating zone, while the crucible containing elemental sulfur was placed outside of the heating zone at a temperature between 180 and 200 °C. The system was flashed with argon gas for 40 min before the furnace was heated. The furnace reached the desired temperature by heating at a rate of 10 °C/min and was kept constant at the desired temperature for 3-9-16-24 and 48 h under the flow of Ar gas at 400 °C and 500 °C. The rate of Ar gas flow was set to 0.15 L per hour. At the end of sulfurization, the furnace was switched off, and the powder was left to cool in the furnace to room temperature. Table 1 summarizes the processing parameters used for the sulfurization process.

2.2. Characterization of powders

X-ray diffraction measurement technique was used to identify the present phases and crystal structures of produced powder samples using a Bruker D2 Phaser. A Scan was performed between $10° \leq 2\theta \leq 90°$ using Cu-Kα radiation of wavelength 1.5406A°. UV–vis spectrophotometry The PG Instruments T92+ test was performed to measure the band gap values of synthesized powders in the wavelength range of 300–900 nm. The surface morphology of the annealed thin films was examined using a Philips XL 30 SFEG SEM with an EDX attachment.

Table 1 – Prcoess parameters for sulfurization process.

Temperature	500 °C	400 °C
Time	24–48 h	3-6-9-16-24 h
Atmosphere	Argon (0.15 lt/h)	
Sulfur Source	Elemental Sulfur	

2.3. Photoelectrochemical measurements

The photoelectrochemical properties of SnS_2 were measured using a Biologic Potentiostat VMP-3 device and a three-electrode electrochemical cell. A platinum wire served as the counter electrode, and a standard calomel electrode was used as the reference electrode. The electrolyte used was an aqueous solution of 0.5 M Na_2SO_4. SnS_2 powders were coated onto FTO glass (1 cm × 1 cm) using the spin coating method to prepare the working electrode. 20 mg of SnS_2 powder was mixed with 2 ml of ethanol and stirred using an ultrasonic stirrer to form a suspension. The coated FTO glass was then heat treated at 100 °C for 1 h to volatilize the solvent and subsequently, annealed at 300 °C for 2 h to stabilize the sample. A solar simulator from ABET Technologies, equipped with a 150 W Xe lamp, was used for photoelectrochemical measurements (chronoamperometry, linear-sweep voltammograms, Mott-Schottky plots, and open circuit potential under dark and illuminated conditions) under 1 sun.

3. Results and discussion

3.1. XRD characterization of SnO_2 and SnS_2 powders

An XRD analysis was carried out after thermal sulfurization to confirm the success of the sulfurization process in the SnS_2 phase. The normalized XRD patterns of pure SnO_2 powders and SnS_2 powders synthesized at 400 °C for 3-9-16 and 24 h, respectively, were given in Fig. 3. The results showed that the success of the sulfurization of the SnO_2 powders increased with increasing sulfurization time. The XRD patterns of

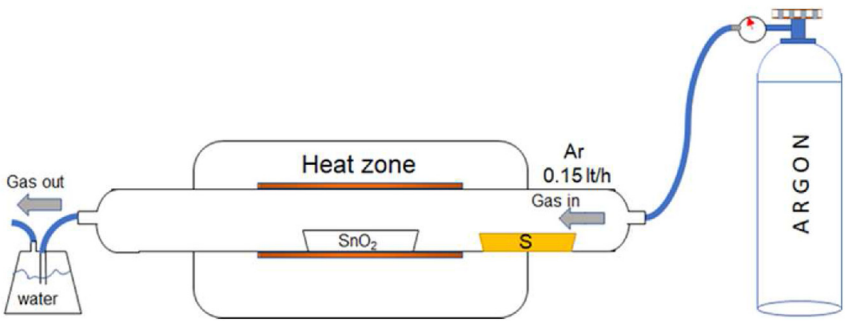

Fig. 2 – Schematic representation of a tube furnace for the thermal sulfurization of SnO_2 powders.

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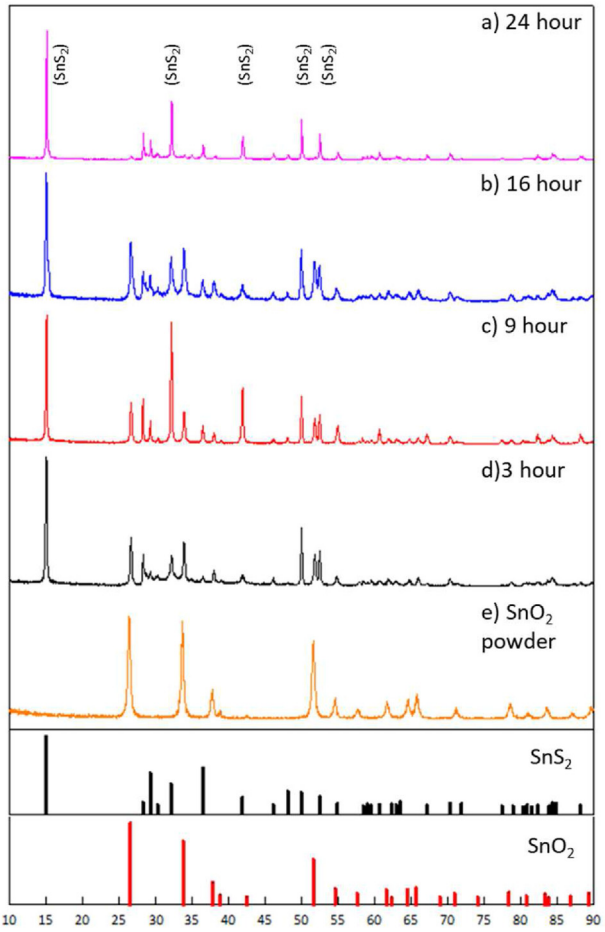

Fig. 3 – XRD patterns e) pure SnO_2 powders and after sulfurization at 400 °C a)24 b)16 c)9 d)3 h.

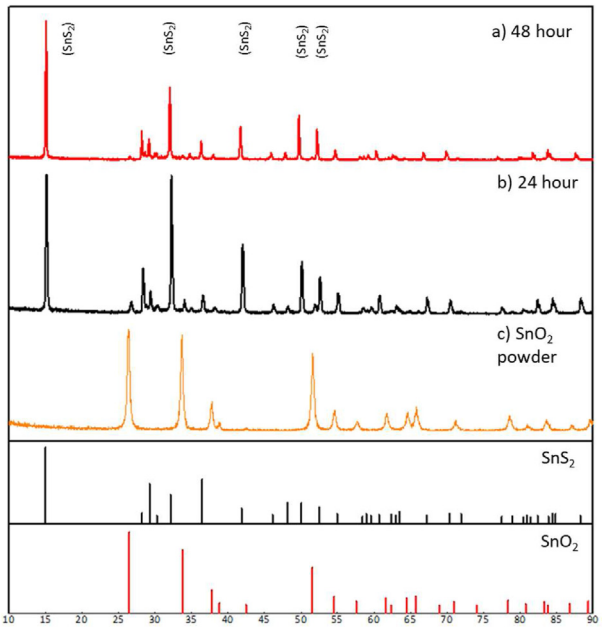

Fig. 4 – XRD patterns c) pure SnO_2 powder and SnO_2 powder safter sulfurization at 500 °C a)24 b)48 h.

samples obtained after sulfurization periods of 24 h revealed minor SnO_2 peaks compared to those of samples acquired after sulfurization times of 3-9-16 h at 400 °C. The results indicated that sulfurization was completed with the appearance of major SnS_2 peaks in the XRD pattern in Fig. 3 (a) after sulfurization for 24 h at 400 °C.

The XRD peaks obtained for the sulfurized samples at 500 °C (Fig. 4). for 24 and 48 h gave almost identical diffraction peaks with the major SnS_2 peaks and minor SnO_2 peaks. The diffraction peaks of sulfurized powder at 400° for 24 h and 500 °C for 48 h clearly proved the existence of a major SnS_2 phase having a hexagonal crystal structure with lattice parameters a = 3,645, b = 3645 and c = 11,802 Aº (PDF 01-089-2357). The major peaks of SnS_2 were detected at 15.13°, 32.13°, 41.87° 50.05° and 52.5°. These peaks correspond to the (002), (102), (104), (110), and (112) planes of hexagonal structure, respectively.

An image of pristine SnO_2 and thermally sulfurized powder at 500 °C for 24 h in an Ar atmosphere can be seen in Fig. 5. Due to the yellow color of SnS_2, sulfurization of SnO_2 powders, white (Fig. 5a), to SnS_2 resulted in powders with yellow tones (Fig. 5b), as expected [19]. The success of sulfurization can be monitored by the change in powder colour. SnS_2 powders with the major phases were successfully produced at 400 °C for 24 h and 500 °C for 24 and 48 h, respectively. The powders synthesized at 500 °C for 24 h and 400 °C for 24 h were further investigated by characterization methods (SEM and UV–Vis absorption spectroscopy).

3.2. *Surface characterization of SnS_2 powders by SEM*

The SEM surface morphology images of the pure SnO_2 powders before and after sulfurization at 400 °C and 500 °C for 24 h were displayed in Fig. 6. Pure SnO_2 (Fig. 6a) has nearly uniform spherical size of powders. In the SEM image of both SnS_2 (Fig. 6b-c) powders obtained by sulfurizing at different temperatures exhibited complete growth of hexagonally shaped flakes as identified by XRD patterns (Fig. 4). In Fig. 6(b and c) seen in the SEM images, the large surface area of hexagonally shaped flakes was believed to allow more efficient light absorption, resulting in increasing the efficiency of the photocatalytic reaction [20]. The grain size of the pure SnO_2 powder was below <100 nm, as can be seen in the SEM images (Fig. 6a), whereas sulfurized powders had a larger grain size. Furthermore, the average size of SnS_2 particles obtained at a temperature of 500 °C was 11 μm, which was larger in comparison with the particle size obtained at 400 °C which was 1 μm. It was evident that as the sulfurization temperature increased, the particle size of SnS_2 powders increased.

To confirm the powder composition, SnS_2 synthesized at different temperatures was analyzed by energy dispersive X-ray (EDX) as shown in Table 2. According to an EDX analysis, before sulfurization, SnO_2 only contained tin and oxygen, and after sulfurization, the atomic ratio of Sn/S elements in SnS_2 powders synthesized at 400 °C and 500 °C, is 1:2.11 and 1:2.07, respectively, which was very close to the theoretical stoichiometry of SnS_2. This result supported the XRD analysis and

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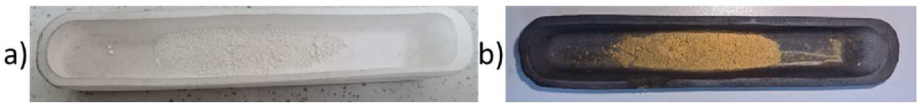

Fig. 5 – Photograph SnO_2 powder a) Before sulfurization b) After sulfurization at 500 °C for 24 h.

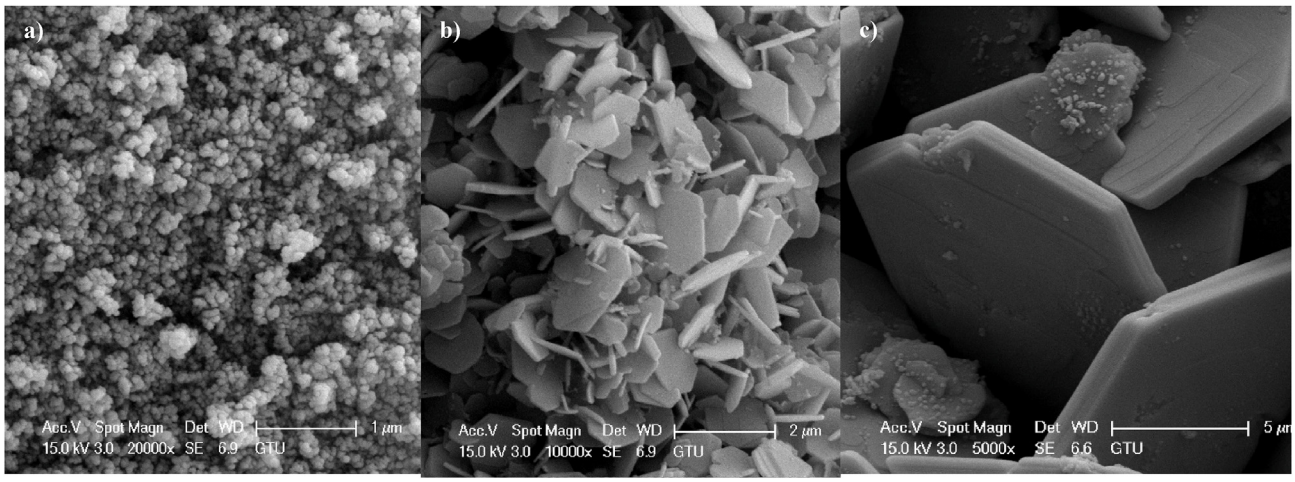

Fig. 6 – SEM Images of SnO_2 powders a) before and after sulfurization for 24 h b) at 400 °C and c) 500 °C.

the experimental findings by showing that the highly purified SnS_2 was effectively produced through thermal sulfurization.

3.3. *Band gap measurement of SnS_2 photocatalyst*

The band gap value (Eg) of the produced SnS_2 powder at 400 °C and 500 °C for 24 h was determined by measuring the powder spectrum absorption using UV–Vis absorption spectroscopy. In Fig. 7, the UV–Vis reflectance spectra and Tauc plot can be seen. The photoactivity of SnS_2 in the visible light region is indicated by the absorption edges observed at a wavelength of 550 nm, as depicted in Fig. 7a. The Eg value of SnS_2 powders was calculated by applying the Kubelka-Munz theory and drawing a Tauc plot using the data obtained from the UV–vis spectrometer measurement [21]. According to the Tauc plot, SnS_2 powder synthesized at 400 °C and 500 °C possessed an indirect band gap of around 2.26 eV and 2.24 eV respectively, which was within the expected range of earlier reported values in the literature [2.28 eV (14), 1.99 eV (15), 2.25 eV (16), 2.22 eV (17), 2.15 (18)]

Table 2 – EDX results after sulfurization at 400 °C and 500 °C for 24 h.

Samples	Elements	Atomic %
SnS_2 – synthesized at 400 °C	Sn	32.09%
	S	67.91%
SnS_2 – synthesized at 500 °C	Sn	32.51%
	S	67.29%

3.4. *Photoelectrochemical characterization results*

PEC results of SnS_2 photoelectrode produced from SnS_2 powders synthesized at 500 °C for 24 h were as follows. When the SnS_2 photoelectrode was illuminated in the neutral electrolyte, the measured open circuit potential (OCP) (Fig. 8a) shifted towards more cathodic potentials. The direction of the measured potential shift indicated that the SnS_2 photoelectrodes had n type semi conductivity, as indicated in the literature [22]. The OCP drop of the SnS_2 photoelectrode was observed as 120 mV in the electrolyte.

Mott-Schottky plot measurements were made to determine the carrier density and the flat band potential of the SnS_2 photoelectrode. The flat band potential of SnS_2 photoelectrodes was calculated by extending the linear portion of the Mott-Schottky plot (Fig. 8b) to the x-axis. The potential value of the flat band was determined to be −0.50 V. In addition, the slope of the Mott Schottky plot was positive, showing that the SnS_2 photoelectrodes had n-type conductivity, with electrons being the majority of carriers. Also, from the slope, charge carrier concentration was calculated according to Equation (1). [$\varepsilon r_{(SnS2)} = 17.7$] [17].

$$N_D = \frac{1.41 \times 10^{32}\left(cm \times F^{-2} \times V^{-1}\right)}{\varepsilon_r \times A^2 cm^4 \times \left(F^{-2} x V^{-1}\right)} \quad (1)$$

Then the charge carrier concentration of the SnS_2 photoelectrode was calculated as $1.49*10^{20}$ cm^{-3} which was higher than the other similar studies [8.48×10^{19} cm^{-3} (14), 5.08×10^{18} cm^{-3} (16)].

The current-voltage response (linear sweep voltammetry) for the SnS_2 photoelectrodes under an applied voltage from

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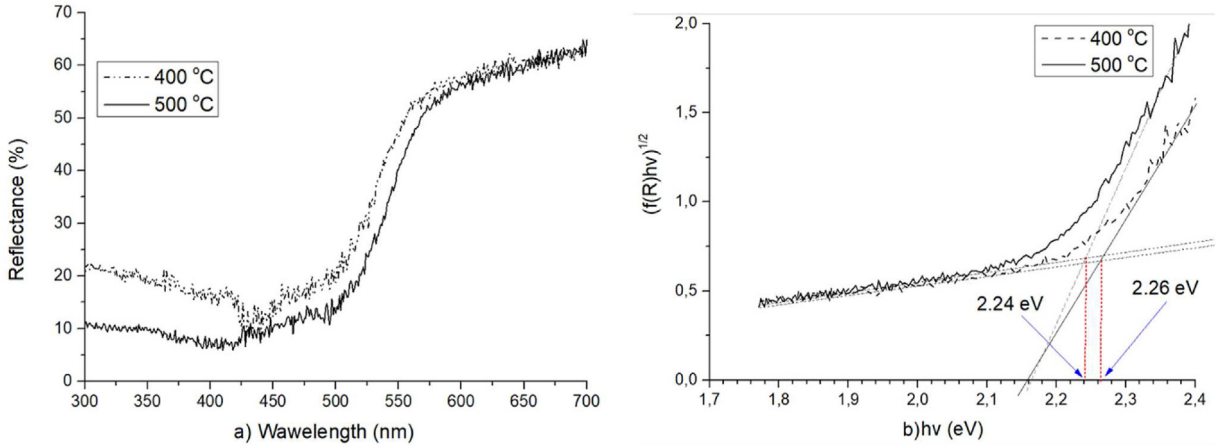

Fig. 7 – a) UV–Vis diffuse reflectance spectra of SnS_2 b) Tauc plot for determine the band gap of SnS_2.

−0.4 to 1.0 V during dark and illuminated conditions.is given in Fig. 8c, demonstrating that a photocurrent was generated even without a bias potential (at OCP potential). This confirms that SnS_2 photoelectrodes are viable photocatalysts, as photoelectrochemical water splitting can occur without any external energy input.

The instantaneous photoresponse of the SnS_2 photoelectrode was investigated using chronoamperometry for both light on and off conditions during 250 s at 0 V vs SCE, (Fig. 8d). The photocurrent density value of the SnS_2 photoelectrode was measured to be 2.5 μA/cm^2. This value is higher than the other values that have been reported in the relevant literature [1 μA/cm^2 (18), 1.5 μA/cm^2 (14), 1.4 μA/cm^2 (15)]. Furthermore, it is noteworthy that certain studies in the literature report higher photocurrents for SnS_2 photoelectrodes; however, it is important to acknowledge that these results were obtained under applied potential conditions. In a study conducted by Liu Y. et al. [16], a photocurrent density of 5 μA/cm^2 was measured for a SnS_2 photoelectrode at 0.5 V (Table 3). It is significant to mention that according to the linear sweep voltammograms (Fig. 8 c)), the measured current density value of around 20 μA/cm^2 which is four times higher than the value of the study conducted by Liu Y. et al. [16], was obtained in our investigation upon applying 0.5 V to the SnS_2 photoelectrode.

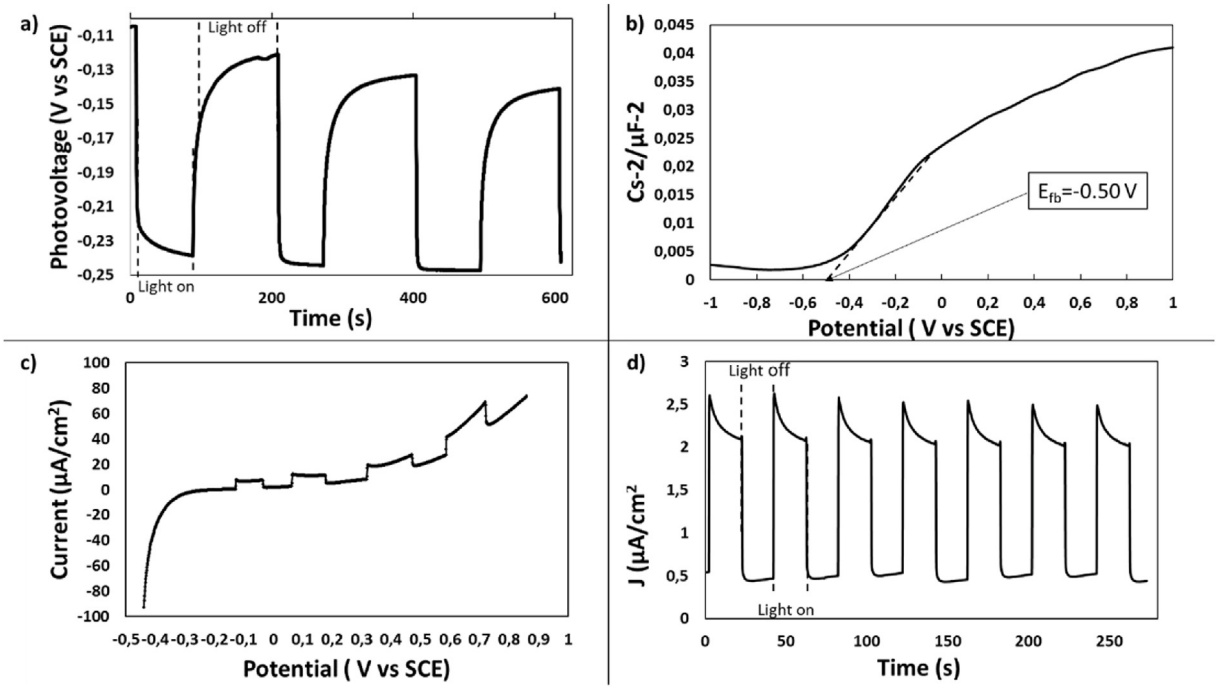

Fig. 8 – PEC Performance of SnS_2 powders after sulfurization at 500 °C for 24 h a) Open Circuit Potential under dark and illuminated conditions. b) Mott-Schottky plots c) Linear-sweep voltammograms from −0.4 V to 1 d) Chronoamerometry at 0 V vs SCE.

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Table 3 – A representative summary of the performance of SnS_2 photoelectrodes stated in the literature.

Synhtesizing Route	Band gap	Solution	Light	Photocurrent	Reference
Thermal Sulfurization	2.24 eV	Na_2SO_4	1000 W/m^2	2.5 μA/cm^2	This work
Hydrothermal	2.28 eV	Na_2SO_4	500 W Xe lamp	1.5 μA/cm^2	14
Hydrothermal	1.99 eV	Na2SO_4	300 W Xe lamp	1.4 μA/cm^2	15
Hydrothermal	2.25 eV	Na_2SO_4	300 W Xe	5 μA/cm^{2v} 0.5 V vs Ag/AgCl	16
Sulfurization from Sn nanoparticles	2.15 eV	Na_2SO_4	300 W Xe	1 μA/cm2	18

Table 3. represents the performances of some SnS_2 photoelectrodes stated in the literature. As shown in the table there are different SnS_2 synthesizing methods.

The band gap values of SnS_2, as reported in the studies mentioned earlier, exhibit minimal variation. The photocurrent density values are observed to be dependent on the experimental conditions. In comparison to similar studies reported in the literature (Table 3), the photocurrent value of SnS_2 (2.5 μA/cm^2) in this particular study exhibits greater performance compared to its counterparts with and without an applied potential.

4. Conclusion

1 The present research examined the photocatalytic characteristics of SnS_2 powders that were produced through thermal sulfurization of SnO_2, indicating the first example of such a study in the literature.
2 SnS_2 powders were successfully synthesized from SnO_2 powders in a tube furnace at 400 °C for 24 h and 500 °C for 24 and 48 h under the Ar atmosphere, and fully sulfurized hexagonal SnS_2 phases were confirmed via XRD analysis.
3 An examination of the powders grain size using a SEM revealed that the powders produced at temperatures of 400 and 500 °C had average grain sizes of 1 μm and 11 μm, respectively.
4 The band gap value of the produced powders was determined as 2.24–2.26 eV as an indirect band gap. With a 2.24–2.26 eV band gap, SnS_2 was an ideal choice for photocatalytic water splitting amongst the metal sulfides.
5 As a result of the photoelectrochemical analysis of the SnS_2 (obtained at 500 °C) photoelectrode produced using SnS_2 powders, it was measured that it has a flat band potential of −0.50 V with a 1.49*10^{20} cm^{-3} charge carrier density (N_d).
6 The photocurrent density value of the SnS_2 photoelectrode was measured to be 2.5 μA/cm^2, higher than the value reported in the literature. This result indicates that SnS_2 photocatalysis is promising photocatalysis as low cost and efficient hydrogen production technique.
7 The thermal sulfurization method was utilized to produce SnS_2 powder, which exhibited a band gap value range similar to that reported in the literature. Also, the PEC characterizations revealed a higher charge carrier density in the SnS_2, and the photoresponse measurements conducted at 0 V vs SCE demonstrated a higher current density comparing with the literature.
8 For a future work, the optimization of sulfurization temperature and time for obtaining the highest efficiency of photocurrent density and hydrogen production will be concluded after PEC characterization of photoelectrodes prepared from SnS_2 powders.

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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