

TiO₂-MoS₂ PAPER BASED MEMBRANE FOR SOLAR EVAPORATION

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Abstract

Clean water scarcity is a critical issue faced by the world. It was caused by insufficient potable water supply and a high cost of water purification systems. As a result, a photothermal evaporator system with the property to absorb light/solar energy and then convert it to heat energy has been rapidly developed. In this work, a solar-driven water evaporation was designed for desalination. The solar evaporator comprises a photothermal molybdenum disulfide-titanium dioxide (MoS₂-TiO₂) composite layer supported onto a cheap filter paper attached. The filter paper was attached to polystyrene foam for support and cotton thread in the centre of the filter paper for water transportation. MoS₂-TiO₂ composite was synthesized and coated onto the filter paper using a simple vacuum-assisted method. Before the solar-driven water evaporation testing, the MoS₂-TiO₂ composite was first characterized using XRD and Raman spectroscopy. The characterization results indicate that MoS₂ introduction into the TiO₂ structure improved the photosensitivity due to the reduction of the bandgap energy from 3.1 eV for TiO₂ to 1.6 eV. The performance of the developed solar evaporator was tested using 35,000 ppm NaCl solution under sunlight for 24 h intermittently. The TiO₂-MoS₂ solar evaporator was observed to have the highest photothermal ability, with 0.807 kg·m⁻²·h⁻¹ evaporation performance and 55.9 % photothermal efficiency compared to other solar evaporators. This indicated that introducing the photothermal MoS₂-TiO₂ layer could enhance

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the solar evaporator performances, making it promising for the desalination application, particularly in impoverished areas with water scarcity.

Keywords: Desalination; MoS₂-TiO₂; photothermal; solar evaporator; solar illumination

Introduction

The growth in water pollution as well as the rise in water demand, have caused a global shortage of freshwater resources. This leads to serious health problems and constraints on global development (Ai *et al.*, 2022). Seawater consists of ~97.5 % of the total earth's surface water, which is the richest water resource on the earth (Guo *et al.*, 2022b). Thus, desalination, which is the removal of salt from water, is the best strategy to overcome the clean water shortage issue. Current desalination technologies such as multi-stage flash distillation (Hasan *et al.*, 2023; Khoshrou *et al.*, 2017), reverse osmosis (Mi *et al.*, 2023; Ridwan *et al.*, 2023), and membrane distillation (Fuzil *et al.*, 2023; Lin *et al.*, 2023) suffer disadvantages such as high maintenance costs, complex construction, and potential for secondary pollution (Cheng *et al.*, 2022a). To deal with the problems, solar desalination is considered the most favourable technology because of its low operating cost and sustainability, as solar energy can be utilized inexhaustibly and is an abundant and environmentally friendly resource to produce fresh water (Guo *et al.*, 2023).

At present, extensive studies on solar evaporation systems have been made and most of them focused on the interfacial solar evaporators as compared to volumetric solar absorption. This is because volumetric solar absorption focuses on heating the bulk liquid which causes it to have lower evaporation efficiency. In interfacial solar evaporators, the heat is produced on the evaporation surface causing it to have a significantly high surface temperature, thus increasing its performance (Mitra *et al.*, 2023).

The interfacial solar evaporator consists of an absorber layer incorporated with photothermal material that possesses high light absorption properties with the ability to convert it to heat energy; and a floating evaporator that is capable of transporting water to the absorber layer (Kospa *et al.*, 2022). The research on developing an effective solar interfacial evaporator primarily focused on the photothermal material and the system design. Various materials were experimented namely carbonaceous materials (Kim *et al.*, 2022; Mitra *et al.*, 2023), metallic

plasmonic materials (Zhao *et al.*, 2015), natural materials (Saleque *et al.*, 2021; Sheng *et al.*, 2020) and polymers (Chen *et al.*, 2018).

For example, Zhu *et al.* (2023) demonstrated that recycled PET film with added carbon black delivered an efficient solar evaporator under sunlight irradiation, achieving a photothermal conversion efficiency of 93.91% and an evaporation rate of 1.457 kg·m⁻²·h⁻¹. Zhang *et al.* (2023) developed coal-based carbon nanomaterial coated on the filter paper's surface that achieved 1.40 kg·m⁻²·h⁻¹ evaporation rate and photothermal conversion efficiency of 90.1%. Molybdenum disulfide (MoS₂) also gains a lot of attention as a photothermal material due to its low bandgap structure, indicating its good photothermal properties (Sang *et al.*, 2023), good chemical stability and low thermal conductivity (Jiang *et al.*, 2022). Xiao *et al.* (2021) developed MoS₂-sodium alginate hydrogel that was wrapped with melamine foam through a low-cost process. It exhibited high performance with 1.92 kg·m⁻²·h⁻¹ evaporation rate in 3.5 wt% NaCl feed solution with 90% photothermal efficiency.

Wang *et al.* (2019) fabricated a carbon-loaded porous titanium dioxide (TiO₂) foam with a high porosity that was able to provide a water transfer pathway with high photothermal conversion property that was able to deliver a high evaporation rate of 1.42 kg·m⁻²·h⁻¹ and 59.43% photothermal. Sang *et al.* (2023) developed a MoS₂/Polydopamine (PDA) composite as a photothermal material as it has excellent light absorbance properties and good wettability. Due to this favourable property, it can deliver 1.51 kg·m⁻²·h⁻¹ evaporation rate and 92% photothermal efficiency.

In this study, a photothermal MoS₂-TiO₂ composite was synthesized for the development of solar absorbers for interfacial solar evaporators because of its high photothermal ability. This evaporator was fabricated by coating the MoS₂-TiO₂ composite on filter paper through vacuum filtration. Both polyethyleneimine (PEI) and PDA were added into the coating solution as PDA had adhesive and photothermal properties while PEI acted as a stabilizer in the formation between the composite and PDA (Chien *et al.*, 2021).

Table 1. The composition of the coating solution and the membrane's thickness

Filter paper	PDA (mg·mL ⁻¹)	PEI (mg·mL ⁻¹)	TiO ₂ (mg)	MoS ₂ (mg)	TiO ₂ -MoS ₂ (mg)	Thickness (mm)
PDA/PEI	2	10	-	-	-	0.190±0.002
TiO ₂	2	10	50	-	-	0.247±0.003
MoS ₂	2	10	-	50	-	0.231±0.008
TiO ₂ -MoS ₂	2	10	-	-	50	0.238±0.006

Materials and Methods

TiO₂ (21 nm $\geq$ 99.55%) and sodium molybdate (Na₂MoO₄·2H₂O, $\geq$ 98%) were purchased from Sigma-Aldrich and l-cysteine (Merck) were used in synthesizing the MoS₂-TiO₂ composite. The Whatman filter papers grade 42 (9 cm in diameter), polystyrene (PS) foam and cotton thread were purchased from a local market. The sodium chloride (NaCl, System) was used as a salt to prepare the feed solution for the photothermal solar evaporator desalination test. All chemicals were used without further purification.

Synthesis of TiO₂-MoS₂ Composite

The preparation of the TiO₂-MoS₂ composite was carried out by using a hydrothermal reaction. First, 600 mg of Na₂MoO₄·2H₂O and 2,500 mg l-cysteine were stirred continuously in 80 mL DI water to dissolve it thoroughly. Then 200 mg of TiO₂ was mixed into the solution and then stirred until homogenous before being transferred into a Teflon-lined stainless-steel autoclave (200°C, 24 h) for hydrothermal reaction. The dark precipitate was centrifuged with DI and dried (60°C, 12 h). The obtained powder was annealed at 800°C under Ar gas for 2 h to obtain TiO₂-MoS₂ composite. To obtain pure MoS₂, similar steps were used without the addition of TiO₂ particles. In the hydrothermal synthesis of MoS₂, the l-cysteine was used as a sulphur precursor while sodium molybdate provide Mo source during the formation of MoS₂ (Mulu *et al.*, 2021; Ramakrishnan and Raina, 2022).

Fabrication of TiO₂-MoS₂ paper-based Membrane

The TiO₂-MoS₂ paper-based membrane was fabricated by adding 0.05 g of prepared TiO₂-MoS₂ into a PDA/PEI solution and stirred and sonicated. The PDA/PEI solution was prepared by firstly mixing 2 mg·mL⁻¹ of PDA in Tris buffer solution (pH = 8.5, 10 mM) and 10 mg·mL⁻¹ PEI. PEI was added in the fabrication of TiO₂-MoS₂ paper-based membrane to act as a dispersion agent to prevent the composite in the coating solution from agglomerating and ensure uniform distribution (Zhu *et al.*, 2007; Yi *et al.*, 2011), while PDA was

used as a coating agent as it has a strong adhesion property for surface coating (He *et al.*, 2022; Tan *et al.*, 2022). PDA also has a broad-spectrum absorption ability and with its hydrophilic properties, it can provide transportation of water to the photothermal layer during the photothermal process (Cheng *et al.*, 2023). The homogeneous coating solution was then vacuum filtered to form a thin TiO₂-MoS₂ layer on the filter paper before being dried at 60°C in the oven. For comparison purposes, the PDA/PEI, TiO₂ and MoS₂ were also coated onto the filter paper using a similar method. Table 1 summarizes the composition of the coating solution used for preparation of paper-based membrane and Figure 1 shows the paper-based membrane prepared in this work.

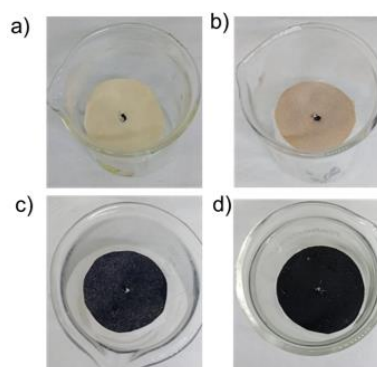


Figure 1. Paper-based membrane for solar evaporator where a) PDA/PEI solar evaporator, b) TiO₂ solar evaporator, c) MoS₂ solar evaporator and d) TiO₂-MoS₂ solar evaporator

Characterization

TiO₂-MoS₂ composite was first characterized using an X-ray diffractometer (XRD- Rigaku) with Cu-K α radiation at 40 mA beam current and 40 kV voltage to determine the crystal structure of the composite. Then, the chemical structure of the TiO₂-MoS₂ composite was evaluated using Raman Spectroscopy (Raman Xplora Plus). To evaluate the composite absorption spectra and band gap, a UV-visible spectrometer (Perkin Elmer Lambda 750) was used for analysis. A contact angle

analysis was carried out using a water contact angle goniometer (AST Products Inc.) system to record and evaluate the wettability of the sample.

Solar Steam Generation Experiment

Solar-assisted evaporator system is composed of TiO₂-MoS₂-coated filter paper supported on the PS foam. The coated filter paper was cut into a circular shape with a diameter of 3.5 cm. The filter paper was attached to the PS foams so that it remains floating on the surface of the water to avoid heat loss to bulk water. The transport of water from the feed water to the evaporation surface on the filter paper was provided by cotton thread through capillary action (Figure 2). The solar evaporator device was placed into a beaker filled with the saline solution containing 3.5% NaCl solution.

The solar evaporator device was illuminated under direct sun. The sunlight illuminated the top part and heated the black photothermal material floating on the saline solution. The whole setup was stabilized for 30 min, and the change in mass was recorded by an electronic balance (Mettler Toledo, ME204). The experimental data were taken every 6 h, and the total time for each test was 24 h. For the data reliability, accuracy and precision, the solar evaporator testing was repeated three times, and the average data were reported. Besides that, the elevation of the surface temperature of the solar evaporator device was recorded using an infrared thermal camera (FLIR E4 Pro, 195 America).

In the evaluation of solar evaporator's performance, the evaporation rate ($\dot{m}$) was calculated using the following equation (Ai *et al.*, 2023; Cheng *et al.*, 2022b):

$$\dot{m} = \frac{\Delta m}{S \cdot t} \quad (1)$$

where Δm represents the mass change of water (kg), S indicates the evaporation area of the solar absorber layer (m²), and t is the irradiation time (h).

The following equation shows the formula to calculate the photothermal efficiency of the evaporator (Kospa *et al.*, 2022):

$$\eta = \frac{H_e \dot{m}}{I} \quad (2)$$

where $\dot{m}$ (kg·m⁻²·h⁻¹) represented the water evaporation rate under light irradiation, I (kW·m⁻²) is the power density of light illumination and H_e is the sensible heat. The calculation of H_e was shown as follows:

$$H_e = C \Delta T + \Delta h \quad (3)$$

where C (4.18 kJ·kg⁻¹·°C⁻¹) represents the specific heat capacity of water, ΔT (°C) is the water temperature difference, and Δh is the enthalpy of water vaporization which is 2,394 kJ·kg⁻¹.

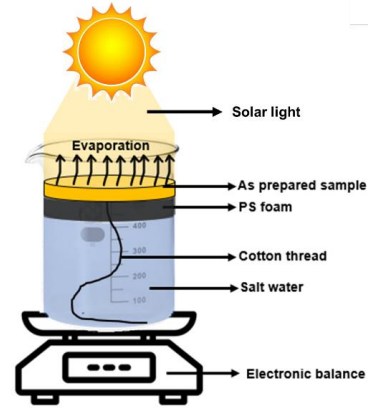


Figure 2. Light-driven evaporator set up evaporator

Results and Discussion

Morphologies and Structural Properties of Composite

Figure 3(a) shows the XRD data of the TiO₂, MoS₂ and TiO₂-MoS₂ composite for comparison. The TiO₂-MoS₂ composite was confirmed through its high distinct diffraction peaks at 2θ values which indicated the peaks for both TiO₂ and MoS₂. The peaks of the MoS₂ composite at 14.42° (002), 33.0° (100), and 58.3° (110) were similar to MoS₂ observed by Jiang *et al.* (2022), Xu *et al.* (2022), and JCPD No. 37-1492 (Zhong *et al.*, 2020). Compared to the JCPDS No. 21-1272 (Li *et al.*, 2022), the diffraction peaks at 25.2° (101), 27.5° (110), and 48.2° (004), 54.1° (105), 55.2° (211), and 63.0° (204) indicate TiO₂ (Carneiro *et al.*, 2014; Fuzil *et al.*, 2023). These results show that the MoS₂-TiO₂ composites were successfully prepared using the hydrothermal method.

Figure 3(b) shows the Raman analysis of the TiO₂-MoS₂ composite and its pure components. MoS₂ shows two peaks at 386 cm⁻¹ and 408 cm⁻¹ which was E_{2g}^1 and A_{1g} vibrational modes of MoS₂, respectively (Yuan *et al.*, 2022). From the comparison between the plane's intensity, A_{1g} vibrational modes were higher than E_{2g}^1 vibrational modes, represent that the MoS₂ were rich of edge sites which is good for photothermal effect (Cheng *et al.*, 2022b). The Raman peak at 145 cm⁻¹ could be

assigned to E_{1g}^1 vibrational mode of TiO_2 component. $\text{TiO}_2\text{-MoS}_2$ shows peaks with same characteristic of TiO_2 and MoS_2 and this indicating the success in the preparation of the $\text{TiO}_2\text{-MoS}_2$ composite. There was a shift of the TiO_2 peaks for

the $\text{TiO}_2\text{-MoS}_2$ composite and this shift was caused by the coupling effect between MoS_2 and TiO_2 (Feng *et al.*, 2019). Both XRD patterns and Raman spectra confirm the formation of the $\text{TiO}_2\text{-MoS}_2$ composite.

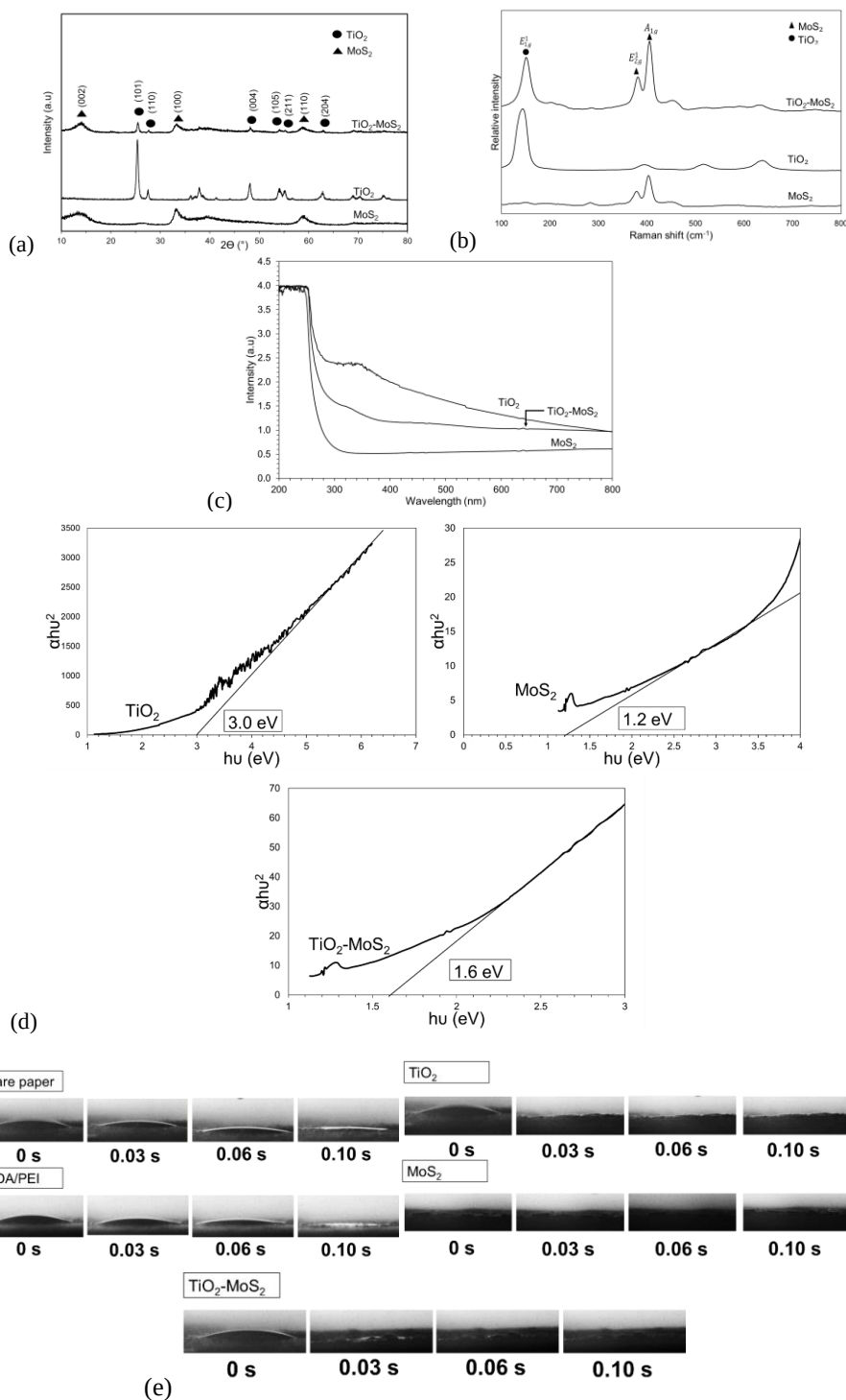


Figure 3. (a) XRD pattern, (b) Raman spectra, (c) UV-Visible absorbance spectra, (d) Tauc plot of MoS_2 , TiO_2 and $\text{TiO}_2\text{-MoS}_2$ composites and (e) The contact angle of the fabricated solar evaporator

The absorbance spectra and band gaps of the TiO₂-MoS₂ composite and its pure components are shown in Figure 3(c) and Figure 3(d), respectively. From the UV-Vis data, the light absorption of TiO₂-MoS₂ composites was much higher compared to pristine MoS₂. This might be able to enhance the photothermal efficiency of the solar absorber. The bandgap of the composite was obtained from the Tauc plot (Figure 3(d)) and the result indicated that the intercalation of MoS₂ with TiO₂ reduced the band gap to 1.6 eV as compared to pure TiO₂ with 3.0 eV. Jarosz *et al.* (2020) stated that lower band gap was more favourable in energy conversion as light, which is photons can be absorbed if their energy is greater than or equal to band gap, leading to the generation of heat. Thus, it is believed that the TiO₂-MoS₂ composites prepared in this work should be able to deliver high light to heat conversion in the solar evaporator when compared to pure MoS₂ as well as pure TiO₂ due to their high light absorption and narrow band gap energy. These criteria would become advantageous for the photothermal ability of solar evaporators.

From Table 1, the modified paper-based membrane solar evaporator was successfully coated homogeneously due to its very low standard deviation value of membrane thickness based on measurements taken at three different spots. This ensured that the solar evaporator produced high overall performance when exposed to sunlight irradiation. From the table, the trend for paper-based membrane thickness was as follows, PDA/PEI < MoS₂ < TiO₂-MoS₂ < TiO₂.

Figure 3(e) displays the wetting properties of the fabricated solar evaporator used in this study. The figure shows that the fabricated solar evaporator was highly hydrophilic, where the water was immediately absorbed when in contact with the filter paper. The bare and PDA/PEI solar evaporators showed a trace of the water droplet that was not fully absorbed after 0.1 s, whereas the water droplet in TiO₂, MoS₂, and TiO₂-MoS₂ solar evaporators was fully absorbed. This suggests that the modification of solar evaporator with TiO₂, MoS₂, and TiO₂-MoS₂ enhance the wettability property of the solar as it could provide a higher water transport to the evaporation interface, causing rapid water evaporation during the solar steam generation (Li *et al.*, 2023; Ye *et al.*, 2023). The performances of the solar evaporator were analysed using 35,000 ppm of NaCl (3.5 wt%) solution to mimic the typical concentration of seawater to evaluate its desalination ability. From Figure 4(a), the performance of the pristine solar evaporator was low where its evaporation rate was $0.217 \pm 0.0048 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ with $14.5 \pm 2.23\%$ photothermal efficiency under sunlight for 24 h

intermittently. This result was lower compared to other studies such as Trinh *et al.* (2023) with an evaporation rate of $0.5 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ and Guo *et al.* (2019) with an evaporation rate of $0.42 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ and 27% photothermal efficiency.

After modification with PDA/PEI, there was a performance improvement where the evaporation rate was $0.508 \pm 0.0152 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ and $34.3 \pm 2.34\%$ photothermal efficiency. The increase in the performance was contributed by the light absorption property of PDA which can enhance the photothermal ability of the solar evaporator (Zhang *et al.*, 2019). Further improvement in solar evaporator was shown by TiO₂ paper-based solar evaporator with $0.510 \pm 0.0131 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ evaporation rate and $34.8 \pm 1.45\%$ photothermal efficiency. Compared to the PDA/PEI paper-based solar evaporator, this higher solar evaporator performance resulted from the broader light absorption range of TiO₂ material (Zheng *et al.*, 2021). The performance of the MoS₂ paper-based solar evaporator with $0.702 \pm 0.0112 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ evaporation rate and $48.0 \pm 3.13\%$ photothermal efficiency was higher compared to the TiO₂ paper-based solar evaporator. This was because of its high photothermal conversion property due to its narrow bandgap structure (Guo *et al.*, 2022a; Jiang *et al.*, 2022) which led to efficient electron excitation and heat generation as there were small energy differences between the bands.

The incorporation of MoS₂ with TiO₂ improved the water evaporation performance of the TiO₂-MoS₂ paper-based solar evaporator where it exhibited the highest performance compared to its other prepared solar evaporator device with an evaporation rate of $0.807 \pm 0.0144 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ and $55.9 \pm 2.56\%$ photothermal efficiency. This improvement was due to the synergic effect of TiO₂ with light absorption ability (Hezam *et al.*, 2023; Mehta *et al.*, 2018; Wang *et al.*, 2019; Wen *et al.*, 2023) and MoS₂ with high photothermal conversion ability (Wang *et al.*, 2021; Xu *et al.*, 2021; Yoo *et al.*, 2022) that can enhance the performance of water evaporation. It suggests that the solar evaporator performance was promising as the photothermal material as shown in previous reports such as the study done by Wu *et al.* (2023) that developed lignocellulose sponge-based evaporators that delivered regeneration rate which could reach $0.63 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ under 1 sun irradiation. Guo *et al.* (2022b) developed MoS₂ bi-layered aerogels with an evaporation rate of water was $1.32 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$ its evaporation efficiency was 86.22% under $1 \text{ kW} \cdot \text{m}^{-2}$ light intensity. Besides that, Jiang *et al.* (2022) fabricated a macroporous MoS₂/PDMS sponge solar evaporator that was able to present an evaporation rate of $1.47 \text{ kg} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$

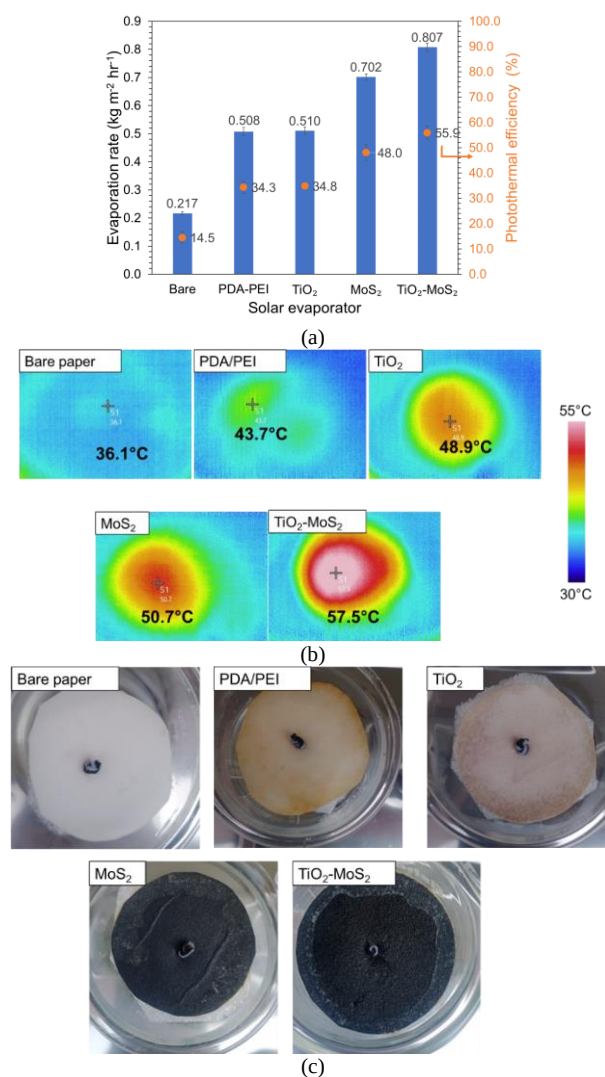


Figure 4. (a) Performances of solar evaporator prepared using different coating types (b) the infrared image TiO_2 , MoS_2 , and $\text{TiO}_2\text{-MoS}_2$ evaporator after 60 min under sunlight irradiation and (c) The image of solar evaporator surface after 24 h of exposure under sunlight

and 81.5% evaporation efficiency under one sun irradiation. Xu *et al.* (2021) fabricated a 2D/2D $\text{Ti}_3\text{C}_2/\text{MoS}_2$ evaporator that achieved $1.36 \text{ kg}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ evaporation rate and 87.2% evaporation efficiency under $1 \text{ kW}\cdot\text{m}^{-2}$ light intensity. Although $\text{TiO}_2\text{-MoS}_2$ paper-based solar evaporator show a high solar absorption and photothermal effect, it shows slightly lower performance compared to other studies and this might be caused by the change of weather in Malaysia.

Figure 4(b) shows the thermal infrared images of the solar evaporator exposed under solar light irradiation for 60 min on 25th July 2023 where the temperature surrounding was 34°C . As a control experiment, the bare solar evaporator showed a very low increment in surface temperature under sunlight

from 34°C to only 36.1°C . In contrast, all the developed solar evaporators exhibited significant temperature increases. The surface temperature increment was following the trend bare paper (36.1°C)<PDA/PEI (43.7°C)< TiO_2 (48.9°C)< MoS_2 (50.7°C)< $\text{TiO}_2\text{-MoS}_2$ (57.5°C). This high surface temperature was due to the solar evaporator's photothermal properties and its set-up that was equipped with PS foam. PS foam was an insulator material and cotton thread allows the water transport to the photothermal layer of the solar evaporator through its wicking effect The $\text{TiO}_2\text{-MoS}_2$ solar evaporator with 57.5°C maximum temperature was comparable to the latest studies such as Zhong *et al.* (2023) where the fabricated polypyrrole decorated cotton fabric based solar evaporator reported

an increase in temperature where its initial temperature was 22.7°C and able to increase to 42°C after exposure under 1 sun irradiation for 60 min. Yan *et al.* (2023) developed a vertical array carbon nanotubes (VACNT) solar evaporator that was capable of raising the temperature to 39.8°C from 22.7°C under 1 sun irradiation for 60 min.

Figure 4(c) portrays the images of a paper-based solar evaporator after 24 h of exposure to sunlight. There was salt accumulation on the solar evaporator indicating that the solar evaporator had a high ability for salt rejection performance. From the figure, there was no salt accumulated on the surface of the bare paper solar evaporator and PDA/PEI paper-based solar evaporator while there was a gradual increase in salt accumulated on the surface of the solar evaporator following the trend TiO₂<MoS₂<TiO₂-MoS₂. Although the TiO₂-MoS₂ paper-based solar evaporator had the highest solar evaporator performance in desalination ability, the increase in accumulation of salt on the surface hindered its performance as the accumulated salt could reflect the sunlight irradiated on the membrane surface (Sun *et al.*, 2023).

Conclusions

The TiO₂-MoS₂ composite synthesized showed better photothermal ability with its lower band gap energy (1.6 eV) and wider light absorption range. The homogenous coating formed on the paper-based membrane surfaces was 0.238±0.006 mm in thickness. The TiO₂-MoS₂ paper-based membrane solar evaporator prepared in the work possessed better light absorption and the photothermal ability to convert light to heat energy, where the solar evaporation rate increased from 0.217 kg·m⁻²·h⁻¹ to 0.807 kg·m⁻²·h⁻¹. Besides that, the photothermal efficiency escalated from 14.5% to 55.9%, while the surface temperature rose to 57.5°C from 34°C after 60 min under sunlight irradiation. The synergic effect of light absorption ability from TiO₂ and superior photothermal conversion ability from MoS₂ enhanced the performance of water evaporation, making it an excellent candidate for solar evaporators. This work suggests new insights into using the photothermal properties of TiO₂-MoS₂ composite for water treatment using renewable energy such as solar.

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