An Efficient Photothermal Conversion Material Based on D-A Type Luminophore for Solar-driven Desalination

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Abstract

Solar-driven interfacial evaporation is a promising technology for desalination. The photothermal conversion materials are at the core and play a key role in this field. Design of photothermal conversion materials based on organic dyes for desalination is still a challenge due to lack of efficient guiding strategy. Herein, a new D (donor)-A (acceptor) type conjugated tetraphenylpyrazine (TPP) luminophore (namely TPP-2IND) was prepared as a photothermal conversion molecule. It exhibited a broad absorption spectrum and strong π–π stacking in the solid state, resulting in efficient sunlight harvesting and boosting nonradiative decay. TPP-2IND powder exhibited high photothermal efficiency upon 660 nm laser irradiation (0.9 W/cm²), and the surface temperature can reach to 200 °C. Then, an interfacial heating system based on TPP-2IND is established successfully. The water evaporation rate and the solar-driven water evaporation efficiency were evaluated up to 1.04 kg/m²·h and 65.8% under 1 sunlight, respectively. Thus, this novel solar-driven heating system shows high potential for desalination and stimulates the development of advanced photothermal conversion materials.

Keywords: tetraphenylpyrazine, solar energy, seawater desalination, photothermal conversion materials, luminophore
TPP-2IND) was prepared as a photothermal conversion molecule. It exhibited a broad absorption spectrum and strong π–π stacking in the solid state, resulting in efficient sunlight harvesting and boosting nonradiative decay. TPP-2IND powder exhibited high photothermal efficiency upon 660 nm laser irradiation (0.9 Wcm⁻²), and the surface temperature can reach to 200 °C. Then, an interfacial heating system based on TPP-2IND is established successfully. The water evaporation rate and the solar-driven water evaporation efficiency were evaluated up to 1.04 kgm⁻²h⁻¹ and 65.8% under 1 sunlight, respectively. Thus, this novel solar-driven heating system shows high potential for desalination and stimulates the development of advanced photothermal conversion materials.

1. Introduction

Although water is one of the most abundant natural resources on Earth, very limited freshwater can be directly used by human beings.¹ Therefore, seawater desalination can alleviate the shortage of freshwater resources.² In addition, it is still challenges that how to access potable water more conveniently, especially for poor regions lack of infrastructure and electricity. Solar energy is the most promising renewable energy among many water evaporation and desalination technologies.³ The effective utilization of solar energy and water resources will effectively reduce carbon emissions, further preserving the environment. Generally, solar interfacial evaporator has much higher evaporating efficiency and heating rate, in which solar-thermal conversion and heating is localized at the air/water interface.⁴ Thus, solar-driven interfacial evaporation is a promising technology to obtain freshwater owing to its high conversion efficiency, industrial application potentiality, and environmentally sustainable development.⁵ However, such evaporation system is a complex coupling process, which is influenced by many factors, such as photothermal conversion materials, heat management and structural design.⁶

Among these factors, the photothermal conversion materials play a key role in the system. So far, there are three popular photothermal conversion mechanisms, including plasmonic localized heating, electron-hole generation and relaxation, and molecular thermal vibration.⁷ Now, the most reported photothermal materials mainly focused on plasma metal nanoparticles,⁸ semiconductor materials,⁹ carbon materials,¹⁰ and polymer materials.¹¹ It is worth noting that organic small molecule based photothermal conversion materials are increasing wide attention since their great potential application in many fields, such as photothermal therapy (PTT),¹² photothermal/photoacoustic (PT/PA) imaging,¹³ and antimicrobials.¹⁴ However, due to the undesirable photobleaching and relatively low photothermal conversion efficiency, there are only a few reports on the research of organic dyes for solar-driven seawater evaporation.¹⁵ Therefore, to achieve excellent photothermal conversion performance and investigate their work mechanism, it is necessary to develop novel photothermal conversion system based on the new organic dyes.

At present, the design strategy of photothermal conversion materials based on organic molecules mainly includes the following aspects: 1) enhancing the absorption in the near infrared region by extending the conjugated length of molecules¹⁶ or covalently connecting electron donor (D) and electron acceptor (A),¹⁷ 2) inhibiting the radiation transition by boosting the quenching effect¹⁸ or generating free radical species,¹⁹ 3) using the charge transfer of organic co-crystals to effectively reduce the energy gap, thereby achieving red-shifted absorption and improving non-radiative transition.²⁰ To now, some D-A structured organic dyes have been utilized for photothermal conversion investigation,²¹ only scattered reports focus on the seawater desalination.²² Therefore, there are large space to verify the rationality and feasibility in the relevant research. To better understand the molecular behavior under light irradiation, much more photothermal materials need to be developed. In this contribution, a new D-A type conjugated tetraphenylpyrazine (TPP) derivative, named as TPP-2IND (Scheme 1), was rationally designed and synthesized. Indeed, the photothermal efficiency of TPP-2IND powder was evaluated upon 660 nm laser irradiation (0.9 Wcm⁻²), and the surface temperature can reach nearly 200 °C. Based on these features, a composite TPP-2IND loading polyurethane (PU) foam was prepared to achieve efficient solar-driven interfacial evaporation. The water evaporation rate and the solar-driven water evaporation efficiency were evaluated up to 1.04 kgm⁻²h⁻¹ and 65.8% under 1 sunlight, respectively. Thus, this work may promote a rapid development of organic photothermal materials for solar-driven desalination.
Scheme 1. The schematic diagram of solar-driven desalination based on organic TPP-2IND.

2. Results and Discussion

2.1 Synthesis and characterization of TPP-2IND

As shown in Scheme S1, the D-A strategy was developed to construct luminophore TPP-2IND. The TPA (triphenylamine) derivative 3 was prepared in a moderate yield from 4-(diphenylamino)phenylboronic acid1 and 5-bromothiophene-2-carbaldehyde 2. When compound 3 was reacted with the bromine reagent NBS (N-bromosuccinimide), the brominated TPA derivative 4 was obtained in 83% yield. Next, 4,4′-dibromobenzil and 1,2-diphenylethylendiamine were utilized as reagents to afford the TPP derivative 5 by dehydration and DDQ (2,3-dichloro-5,6-dicyano-1,4-benzoquinone) oxidation. Boric acid ester-substituted TPP derivative 7 was synthesized by Miyaura borylation, and then hydrolysis reaction was conducted to afford TPP derivative 8 with much higher reactivity. As expected, TPP derivative 9 was obtained by Suzuki coupling between TPP derivative 8 and TPA derivative 4. Finally, the desired TPP-2IND was afforded by Knoevenagel condensation. Their structures were characterized by $^1$H NMR, $^{13}$C NMR and high resolution mass spectrometry (HRMS). The detailed synthetic route and characterization are presented in Scheme S1 and Figure S1-S14 in the Supporting Information.

2.2 Photophysical and photothermal properties of TPP-2IND

The photophysical properties of TPP-2IND were explored in various solvents. As depicted in Figure 1A, the UV-vis absorption spectra of TPP-2IND were recorded in toluene, dichloromethane (DCM), tetrahydrofuran (THF), ethyl acetate (EtOAc), chloroform (CHCl$_3$), acetone, and dimethyl sulfoxide (DMSO), exhibiting similar absorption profiles and affording a strong absorption band around 370 nm in dilute solutions ($5 \times 10^{-6}$ M), which mainly originated from the $\pi$-$\pi^*$ transition of the large conjugated molecular backbone. There existed another broad absorption bands in long-wavelength region (500-550 nm), which was assigned to the intramolecular charge transfer (ICT) from electron donor-acceptor structure.$^{[22]}$ As illustrated in Figure 1B, the photoluminescence (PL) wavelength underwent an obvious red shift from 595 nm to 693 nm upon increasing the solvent polarity from toluene to DMSO. It should be noted that the fluorescence intensity increased initially and decreased afterwards as the solvent polarity increased (Figure 1C).

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The PL spectra of TPP-2IND also showed a typical twisted intramolecular charge transfer (TICT) effect in THF/water mixture due to molecular push-pull electronic structure. In pure THF solution, TPP-2IND had an emission maximum at 620 nm and exhibited intense red fluorescence (Figure 2A). Meanwhile, a slight red-shift could be observed during this process. With the addition of polar solvent of water, the fluorescence intensity of the mixture has dramatically decreased (Figure 2B). When more water was added, obvious molecular aggregates was observed in the filter cake by filtering the solution. TICT effect was reduced to some extent, and therefore the weak fluorescence was mainly attributed to aggregation-caused quenching (ACQ). Thus, its excited state energy was released by non-radiative transition, which means that the possibility of photothermal transformation is enhanced.

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As expected, TPP-2IND exhibited a broad absorption spectrum in the solid state (Figure 2C), effectively promoting the absorption of sunlight. Meanwhile, the non-radiative decay process was significantly enhanced upon aggregation. A moderate fluorescence quantum yield ($\Phi_F$) of 25% was obtained in pure THF solution, while its $\Phi_F$ remarkably decreased to 7% in solid state (Figure 2D). This phenomenon could be explained by the intermolecular $\pi-\pi$ stacking interaction between the aromatic rings, leading to an obvious decrease of fluorescence quantum yield in the aggregated state. (24) As presented in Figure S15, the fluorescence decay curve of TPP-2IND revealed that its lifetime is 0.61 ns in solid state. Based on quantum chemical calculation (detailed calculation process is presented in Supporting Information), $\Phi_F$ can be expressed as $\Phi_F = k_r/(k_r + k_{nr} + k_{isc})$. $k_r$ and $k_{nr}$ are the radiative and nonradiative decay rate constant respectively, $k_{isc}$ is the intersystem crossing rate constant from the singlet state ($S_1$) to triplet state ($T_1$). Generally, the spin-orbital coupling (SOC) constant between $S_1$ and $T_1$ is small in pure organic molecules, the intersystem crossing process from $S_1$ to $T_3$ is negligible. Consequently, $\Phi_F$ can be simplified as $\Phi_F = k_r/(k_r + k_{nr})$. Thus, $k_r$ and $k_{nr}$ are calculated to be $1.15 \times 10^8$ s$^{-1}$ and $1.53 \times 10^9$ s$^{-1}$, respectively. The smaller $k_r$ and larger $k_{nr}$ of TPP-2IND confirm that the strong D-A interaction results in an acceleration of non-radiative relaxation pathway, and a sharp decrease of PL efficiency in the aggregated state.

Theoretical calculations were carried out to investigate the fluorescent behavior. As displayed in Figure 3A, the highest occupied molecular orbital (HOMO) was mainly distributed on the electron-donating triphenylamine (TPA) backbone, while the lowest unoccupied molecular orbital (LUMO) was predominantly concentrated on the electron-withdrawing 1,3-indandione moiety and thiophene skeleton, indicating strong D-A interaction. The energy gap of the LUMO and the HOMO was 2.459 eV. It has been confirmed that spatial separation of HOMO and LUMO is beneficial to attain small $\Delta E_{ST}$ and efficient ROS generation. As a consequence, the total reactive oxygen species (ROS) generation efficiency was evaluated by ROS indicator 2',7'-dichlorodihydrofluorescein diacetate (DCFH-DA). The emission intensity of DCFH-DA showed a 24-fold PL enhancement after white light irradiation, proving an obvious ROS generation of TPP-2IND (Figure 3B). To further confirm the species of ROS, a commercial $^{1}O_2$ indicator 9,10-anthracenediyb-bis(methylene)-dimalonic acid (ABDA) was used to monitor the absorption changes under white light irradiation. As depicted in Figure 3C, 12% of ABDA was consumed in the presence of TPP-2IND, meaning low generation efficiency of singlet oxygen, while the absorbance of free ABDA remained almost unchanged. Thus, it is speculated that TPP-2IND was able to produce abundant type I ROS against hypoxic environment. Then
dilhydrorhodamine 123 (DHR123) was involved as free radical ROS indicator to detect $\text{O}_2^*$. As revealed in Figure 3D, the PL intensity of DHR123+ TPP-2IND solution increased sharply with light irradiation, revealing the efficient generation of type I (free radical-dominated) ROS.

Figure 3. (A) Optimized molecular geometry and electron cloud distributions of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) of TPP-2IND, which were calculated by B3LYP-D3/6-31G$.^*$. (B) ROS generation of TPP-2IND (10 $\mu$ M) upon white light irradiation using DCFH-DA as an indicator. (C) decomposition rates of ABDA (for $^1\text{O}_2$ detection) in the presence of TPP-2IND (10 $\mu$ M) upon white light irradiation for different times. (D) DHR123 (for $^\bullet\text{O}_2$ detection).

The photothermal properties were then investigated by UV-vis spectra. TPP-2IND powders exhibited a broad absorbance from UV to visible region (Figure S16). Therefore, TPP-2IND is a viable organic dye for solar thermal conversion. The photothermal property of TPP-2IND was real-time monitored under infrared thermal imager. As depicted in Figure 4A, 50 mg of TPP-2IND powders were irradiated by 660 nm laser (0.9 W cm$^{-2}$), and the surface temperature increased sharply from room temperature to nearly 200 °C in 20 s, then cooled naturally after switching the laser off, the temperature returned to original state, showing rapid photothermal conversion during this process. Next, the influence of irradiation power density on the photothermal conversion was investigated from 0.5 to 0.9 W cm$^{-2}$ using 5 mg of TPP-2IND powders (Figure 4B). Obviously, the higher power density was applied, the higher heating rate and maximum equilibrium temperature could be observed. Then, the thermal cycling stability was evaluated upon laser irradiation turned on and off for eight times (Figure 4C). It should be noted that the equilibrium temperature was highly related to the irradiation power density (Figure S17), indicating a satisfactory photothermal conversion efficiency of TPP-2IND. The X-ray diffraction (XRD) results indicated that TPP-2IND loosely packed in the disordered amorphous state (Figure S18), which favors nonradiative relaxation pathways. Moreover, the thermogravimetric analysis (TGA) showed that the decompose temperature of TPP-2IND was above 300 °C (Figure S19), exhibiting high thermal stability of the molecule.

Figure 4. (A) IR thermal images of TPP-2IND powder under 660 nm laser irradiation (0.9 W cm$^{-2}$) and then turned off. (B) IR thermal images of TPP-2IND powder (5 mg) under 660 nm laser irradiation and then turned off. (C) Anti-photobleaching property of TPP-2IND powder during eight cycles of heating–cooling processes under 660 nm laser irradiation (0.8 W cm$^{-2}$).

2.3 Solar-Driven Water Evaporation

Based on the above photothermal performance, TPP-2IND was introduced into the porous PU foam with low thermal conductivity to form an interfacial solar-driven water evaporation system. First, the solution was prepared by dissolving different mass of 5-50 mg of TPP-2IND in dichloromethane (Figure S20). Then, the PU foam with different contents of TPP-2IND (5, 10, 20, 30, 40 and 50 mg) were obtained by impregnation process and drying at 80 °C for 1 h. From the SEM images we can see that the PU surface became rough gradually with the increase of the loading amounts of TPP-2IND, indicating TPP-2IND could be effectively loaded onto the blank PU foam (Figure S21). The higher loading content of TPP-2IND can enhance the photothermal conversion performance to some extent. The criss-crossed microporous structure inside the foam effectively promoted the transport of water and localized the heat generated by TPP-2IND, greatly accelerating the evaporation of water. In contrast to blank PU foam, PU+TPP-2IND exhibited higher
UV-vis light absorption properties, implying that PU foam contributed slightly to photothermal conversion (Figure S22 and S23). Meanwhile, the thermal conductivity of blank PU and PU+TPP-2IND was evaluated to be 0.03137 and 0.03749 W m$^{-1}$ K$^{-1}$ at 25 °C, respectively. And the thermal conductivity increased to 0.03907 W m$^{-1}$ K$^{-1}$ as the temperature increased from 25 to 55 °C (Figure S24), demonstrating a low thermal conductivity. Therefore, the low thermal conductivity of PU+TPP-2IND is benefit for localized heating at the interface and further enhancing solar vapor generation.\[28\]

Then, the solar photothermal conversion of TPP-2IND loaded foams were estimated by recording the temperature variation upon simulated sunlight irradiation (1 sun, 1 kW m$^{-2}$). The real-time temperature on the surface quickly increased from room temperature to an equilibrium within 60 s (Figure S25A). Furthermore, the equilibrium temperature could be reached higher plateau with the increase of the loading amount of TPP-2IND. As depicted in Figure 5A and Figure S25B, the mass change of water under PU+TPP-2IND foams with different loadings were measured under 1 sunlight irradiation to investigate the efficiency of solar-driven water evaporation rate. The equilibrium temperature of the blank PU foam was maintained at 27 °C, which means that PU foam had barely no contribution to photothermal conversion (Figure 5B). In sharp contrast, the equilibrium temperature of PU+TPP-2IND foam with 40 mg of TPP-2IND loading can reached to 76°C under the same condition. Among them, the loading content of 40 mg is the optimal loading of TPP-2IND. It should be noted that such high temperature is attributed to the low thermal conductivity of the PU+TPP-2IND, as well as the porous structure for localizing thermal energy. Meanwhile, the IR thermal images were taken by IR thermal camera and showed the distinct temperature changes of PU+TPP-2IND foam and the blank PU foam, exhibiting a rapid photothermal conversion (insets of Figure 5C).

**Figure 5.** (A) The simulated setup used to record the water mass change under solar irradiation. (B) Photothermal conversion behavior of PU and PU+TPP-2IND-40mg foams under 1 sun irradiation (1 kWm$^{-2}$). Inset shows IR thermal images of PU and PU+TPP-2IND foams under 1 sun irradiation. (C) The temperature changes of PU and PU-TPP-2IND-40mg foams floating on water against sunlight irradiation time. Insets show the photos under simulated sunlight and IR thermal camera upon 1 kWm$^{-2}$. (D) Water evaporation curves with PU foam and PU-TPP-2IND-40mg foam under simulated sunlight with an intensity of 1 kWm$^{-2}$. The amount of PU-TPP-2IND used in preparing PU-TPP-2IND foam is 40 mg. Room temperature is about 26 °C. (E) The measured concentrations of four primary ions (Na$^+$, Mg$^{2+}$, Ca$^{2+}$, and K$^+$) in an simulated seawater sample before and after desalination.

The PU+TPP-2IND foam can float on the water surface and be used to heat water for steam generation under sunlight irradiation (Figure 5C). To transform the absorbed sunlight to thermal energy, most of the solar energy is used to drive liquid-vapor phase change by solar evaporators, and the less energy is lost to the bulk water and dissipate into the environment. However, the heat conduction loss to the bulk water accounts for the largest proportion in the total heat loss.\[29\] Benefiting from the low thermal conductivity of PU+TPP-2IND foam itself, the IR thermal photo exhibited that the converted energy was predominantly localized in the floating PU+TPP-2IND, which means that the converted energy can be directly transferred to the small amount of water, thus improving evaporation efficiency. The surface temperature between PU+TPP-2IND foam and water surface was significantly higher than that of PU foam (Figure 5C and S25B). Under 1 sun irradiation, the equilibrium temperature of PU-TPP-2IND foam loaded with 30 mg TPP-2IND was 45 °C, while the equilibrium temperature of the blank sample PU was only 31°C. Compared to the bulk water temperature (26°C), the surface temperature of PU+TPP-2IND foam represented a 19 °C increment, thus greatly promoting the water evaporation speed according to the previous reports.\[27,30\]

With the loading of TPP-2IND increasing from 5 to 10, 20, 30 and 40 mg, respectively, the water evaporation rate increased from 0.57, 0.58, 0.72, 0.89 and 1.04 kgm$^{-2}$h$^{-1}$, respectively (Figure 5D and Figure S25C).

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Compared with the slow water evaporation rate of pure PU foam (0.42 kg m\(^{-2}\) h\(^{-1}\)), the water evaporation efficiency of PU foam with 40 mg of TPP-2IND loading was increased by nearly 2.5 times, confirming that PU+TPP-2IND foam could be used for efficient water evaporation. Then the solar-driven water evaporation efficiency (\(\eta\)) for PU+TPP-2IND foams were calculated (Figure S25D), and the results showed that the efficiency of PU loaded with 40 mg of TPP-2IND can reach up to 65.8% (detail calculation process is shown in Supporting Information). In addition, relatively stable maximum temperature was observed for TPP-2IND and PU+TPP-2IND foam during the continuous irradiation for 2 h (Figure S26A and B). The UV-vis spectra were in accordance with the initial state after long-time laser irradiation for 2 h (Figure S26C), illustrating its high stability against photobleaching.

To further investigate the solar desalination capability of the PU+TPP-2IND, simulated seawater was prepared to investigate desalination ability by using simple solar evaporator (detail preparation method of simulated seawater is shown in Supporting Information). The primary ions concentrations of Na\(^+\), Mg\(^{2+}\), Ca\(^{2+}\) and K\(^+\) in simulated seawater before and after desalination were determined by inductively coupled plasma optical emission spectroscopy (ICP-OES). As displayed in Figure 5E, the results showed that the concentrations of the distilled water were reduced by 3 or 4 orders of magnitude lower than the bulk water, and below the potable water standard of the World Health Organization (WHO) and the US Environmental Protection Agency (EPA), showing good performance for potential seawater desalination.

3. Conclusion

In this work, a new D-A type conjugated molecule TPP-2IND has been successfully prepared for solar-driven interfacial evaporation. It exhibited a broad absorption spectrum and strong \(\pi-\pi\) stacking in the solid state, resulting efficient sunlight harvesting and boosting nonradiative decay. As expected, TPP-2IND exhibited rapid photothermal conversion and high thermal stability under 660 nm laser irradiation. Thus, a composite TPP-2IND-loading PU foam was successfully fabricated to establish an interfacial heating system. The water evaporation rate and the solar-driven water evaporation efficiency were evaluated up to 1.04 kg m\(^{-2}\) h\(^{-1}\) and 65.8% under 1 sunlight, respectively. Considering the increasing demand of photothermal materials, this material based on organic molecule provided a practicable approach for solar thermal conversion and applications.

4. Experimental Section

Materials: All the reagents and solvents used were purchased from Energy Chemical, and used without further purification. \(^1\)H NMR and \(^{13}\)C NMR spectra were measured on an Agilent Technologies 400 MHz spectrometer. High-resolution mass spectrometry (HRMS) was recorded on Japan Shimadzu LCMS-IT-TOF.

Steady-state spectral measurements: Photoluminescence (PL) spectra was measured on HJY fluorescence spectrophotometer (France).

Photothermal characterizations measurements: The 660 nm laser beam was generated by a LR-MFJ-660 laser (Changchun Lirui Optoelectronic Technology Co.). And the temperature response of the sample was measured with an IR thermal camera (FLIR-E6390).

Desalination of seawater: Our system uses laboratory simulated seawater samples for desalination. The concentrations of all the four primary ions (Na\(^+\), Mg\(^{2+}\), Ca\(^{2+}\) and K\(^+\)) originally present in the sea water were examined by inductively coupled plasma spectroscopy (ICP-OES, Agilent 5110) before and after desalination.

Long-term durability: We carried out the long-term stability to check the durability in this laboratory simulated seawater evaporation. The cycle performance of PU+TPP-2IND evaporation system under fixed optical illumination (1sun) was investigated under air, with each cycle sustained for over 1 h. The mass change was recorded every 1 min. During water evaporation process, the temperature of the surface of PU+TPP-2IND foam was monitored by IR camera to study the photostability of this TPP-2IND-loaded PU.

Thermal conductivity measurement: The thermal conductivity of pure PU and PU+TPP-2IND foams were
measured using a hotdisk (TPS2500S). A hot-wire method was employed to measure thermal diffusivity of materials.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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