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Bioinspired Bifunctional Membrane for Efficient Clean Water Generation

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ABSTRACT

Solving the problems of water pollution and water shortage is an urgent need for the sustainable development of modern society. Different approaches, including distillation, filtration, and photocatalytical degradation, have been developed for the purification of contaminated water and the generation of clean water. In this study we explored a new approach in using solar light for both water purification and clean water generation. A bifunctional membrane consisting of a top layer of TiO$_2$ nanoparticles (NPs), a middle layer of Au NPs, and a bottom layer of anodized aluminum oxide (AAO) was designed and fabricated through multiple filtration processes. Such a design enables both TiO$_2$ NP-based photocatalytic function and Au NP-based solar-driven plasmonic evaporation. With the integration of these two functions into a single membrane, both the purification of contaminated water through photocatalytical degradation and the generation of clean water through evaporation were demonstrated using simulated solar illumination. Such a demonstration should also help open up a new strategy for maximizing solar energy conversion and utilization.
INTRODUCTION

With the accelerated development of modern society and rapid population growth, the consumption of energy increases significantly every year, which in turn leads to severe environmental pollution problems, especially air and water pollution. Over-discharging of contaminants into natural water resources leads to the shortage of clean water globally.\textsuperscript{1,2} It is predicted that by the year 2025, nearly two-thirds of the world’s population will live in water-stressed countries.\textsuperscript{3} One way to battle water shortage is to reuse contaminated water through various de-contamination processes, such as purification or distillation. Due to the cleanness and sustainability of solar energy, using solar energy for clean water regeneration has attracted significant attention, and tremendous progress has been reported recently.\textsuperscript{4-7}

One promising and widely studied route to use solar energy for the purification of water is decontamination through photocatalytic processes. In the photocatalytic decontamination process, a photoactive material absorbs sunlight and creates highly active species, such as H$_2$O$_2$, OH$,^\cdot$, O$_2^-$, and O$_3$, which can react with contaminants to produce small nontoxic molecules.\textsuperscript{7} Most of the catalysts studied, however, can only be activated by specific ranges of photon wavelengths, while the other wavelengths of the solar spectrum are not efficiently used. For example, titanium dioxide (TiO$_2$), which has received the most attention among all the photocatalytic materials studied in the removal of highly toxic and non-biodegradable pollutants in water,\textsuperscript{8,9} has a narrow light absorption band in the near-UV region. This narrow absorption band contributes to the low photocatalytic efficiency of TiO$_2$.\textsuperscript{10} There have been many studies on the modification of structure and morphology,\textsuperscript{11,12} surface chemistry,\textsuperscript{13-15} and composition\textsuperscript{16-18} of TiO$_2$ in order to enhance its photocatalytical efficiency. In addition to increasing the range of the absorption through materials engineering of catalysts, expanding the usage of photons in the range outside of the catalytic absorption band will also lead to
an increased efficiency of solar energy utilization. Even within the absorption band, some of the absorbed photons are converted into heat\cite{8} and approaches that take advantage of such heat can also help improve the overall efficiency in solar energy usage.

Another promising route for the use of solar energy for water purification is the solar-driven water evaporation process. In photocatalytic reactions, solar energy is converted into chemical energy and induces the degradation of contaminants, while in solar-driven water evaporation processes the solar energy is converted into thermal energy, which in turn drives efficient water evaporation.\cite{19,21} Solar-driven water evaporation has attracted increased attention lately.\cite{22,23} In particular, plasmonic photo-thermal conversion was demonstrated to be a highly efficient approach for solar-driven water evaporation.\cite{24,25} We recently developed a plasmonic interfacial evaporation system that was inspired by the localized heating effects of biological systems.\cite{26,28} In this bioinspired interfacial evaporation system, a gold nanoparticle (Au NP) film fabricated through either self-assembly or filtration floats at the air-water interface. Under the solar irradiation, the plasmonic Au NP film is heated, and generates a localized “hot-zone” at the air-water interface. This confined heating effect can maximize the energy efficiency of the water evaporation since it avoids the non-necessary heating of the bulk liquid.

In this work, we intend to combine solar-driven evaporation with photocatalytic reactions to expand the usage of solar energy that is not used by the photocatalysts. Specifically we would like to demonstrate a bifunctional membrane system, with the intent of maximizing the usage of solar energy and further increasing the efficiency of water purification. Nature already provides examples of bifunctional systems that utilize solar energy for both catalytic reaction and water evaporation. The most typical examples include plant leaves. Plant leaves absorb and convert solar energy into
chemical energy through photosynthesis process. Besides the function of photosynthesis, plant leaves also have the function of transpiration, in which the leaves use solar energy to drive surface water evaporation, and therefore facilitate the transportation of nutrients from the plant root to other parts of the plant. This evaporation process also helps cool the plant. Inspired by the multifunctionality of plant leaves for solar energy utilization, we designed and fabricated a bifunctional membrane system that combines both the solar-driven catalytic degradation of water contaminants and solar-driven water evaporation for the purification of water. The membrane (TiO$_2$-Au-AAO membrane) is composed of a photocatalyst layer of TiO$_2$ NPs, a plasmonic photon-to-heat converting layer of Au NPs, and a supporting anodized aluminum oxide (AAO) layer (Figure 1). Upon simulated solar light irradiation, the photocatalytic function of the membrane degrades the contaminants in the water for the generation of purified water; the evaporation function of the membrane through localized heating can generate clean water vapor and therefore purified water after condensation. Through these two simultaneous processes, water with different degrees of purities could be produced at the same time and used for different applications. This bioinspired bifunctional membrane offers an alternative approach in solar-driven clean water generation, and will help improve the efficiency in solar energy usage.
Figure 1. The bifunctional TiO$_2$-Au-AAO membrane has three layers: a top TiO$_2$ NP layer, a middle Au NP layer, and a bottom AAO layer. This design integrates both photocatalytic degradation and solar vapor generation functions into a single system for the purification of contaminated water.

EXPERIMENTAL SECTION

Materials

Chloroauric acid (HAuCl$_4$, 49~50% Au basis), trisodium citrate dihydrate, hydroxylamine hydrochloride and Rhodamine B (RhB) were obtained from Aladdin (Shanghai, China). All the materials were analytical grade and used without further purification. Titanium dioxide (TiO$_2$, anatase, ~25 nm in diameter) was also purchased from Aladdin. Anodic Aluminum Oxide (AAO) membranes were purchased from Lessonnano Technology Co. Ltd. (Guangzhou, China) with pore size of ~20 nm. Deionized water was produced by Millipore Water Purification System (NANO pure, Billerica, MA, USA, 18.2 MΩ).
Fabrication of Bifunctional Membrane

Au NPs were synthesized using the citrate reduction method.\textsuperscript{29,30} First, a 50-mL aqueous solution of 1 mM HAuCl\textsubscript{4} was injected into a 100-mL round-bottom flask attached with a condenser and brought to boil under vigorous stirring. Next, a 5-mL solution of 38.8 mM sodium citrate was added into the flask and the color of the solution immediately changed from pale yellow to reddish-purple. After boiling for 10 min, the solution was cooled down and stirring was continued for an additional 15 min. The as-synthesized colloidal Au NPs were further grown into larger particles through the addition of 1.25-mL 0.2 M hydroxylamine hydrochloride and 1.5-mL 1\% HAuCl\textsubscript{4} at room temperature to the flask. After 35-min of reaction, Au NPs with a diameter of ~ 50 nm were obtained and the particle solution was stored for later use.

To construct the bifunctional TiO\textsubscript{2}-Au-AAO membrane, first ~25-mL as-synthesized 50-nm Au NPs solution was filtered through an AAO membrane and dried in air. Next, a 0.5-mL solution of 25-nm TiO\textsubscript{2} NPs (1.0 mg/mL, dispersed in DI water through sonication) was filtered through the Au NP coated AAO membrane to form a photoactive layer on the top. This composite membrane was dried and stored for the later property testing. Two types of control samples that contain either a Au NP film only or a TiO\textsubscript{2} NP film only on top of the AAO membrane were also prepared using the same filtration method with same amount of corresponding NP solutions.

Characterizations

The morphology and size of the synthesized Au NPs were observed with a TEM (Tecnai G2 Spirit Biotwin, FEI). The bifunctional TiO\textsubscript{2}-Au-AAO membrane samples were examined by field-emission scanning electron microscopy (SEM, FEI Sirion 200, 5kV). The optical absorption spectra of the samples in the range of 200–800 nm were obtained by an UV-VIS-NIR spectrometer.
(PerkinElmer, Lambda 750S). The optical absorbance measurement for RhB solution was performed using an Ocean Optics spectrometer (HR2000+CG, Ocean Optics, USA) attached with two optical fibers (QR400-7-Vis-NIR fiber, Ocean Optics, USA). A 6W tungsten-halogen light source (HL-2000, Ocean Optics, USA) was also used for the measurement.

**Photocatalytic Activity Test**

To evaluate the photocatalytic property of the fabricated bifunctional membrane, an organic dye RhB was used as the model material and its photodegradation processes by both the bifunctional membrane and the control membrane samples were studied. For a typical test, an aqueous RhB solution (20 mg/L) was prepared and 10-mL of the solution was injected into a 15-mL quartz beaker. The membrane sample was inserted and floated on top of the RhB solution. Before exposure to simulated solar light, the mixtures were kept in the dark at room temperature for 12 h to reach adsorption–desorption equilibrium. A 300W Xe lamp (Shanghai Bilon Instrument Co., Ltd) equipped with a Fresnel lens (Shenzhen Salens Technology Co., Ltd.) was used as the light source. After pre-conditioning, the quartz beaker with RhB solution and membrane sample was placed at the focal position of the Xe lamp and irradiated by the Xe light from the top. The light intensity was maintained at ~2.1 W during the irradiation, and the absorbance spectra of the sample solution were recorded every 30 min using the Ocean Optics spectrometer. The concentration of RhB was determined by the absorbance at 553 nm. The following equation was used to calculate the degradation degree ($D_e$) of RhB:

$$D_e = \frac{(C_0 - C)}{C_0} \times 100\%$$

where $C_0$ is the initial concentration of RhB and $C$ is the concentration at the irradiation time of $t$.

Due to the evaporation of water during the photocatalytic testing, the concentration of the RhB was
calibrated according to the weight loss of the solution. After finishing each test, the membrane samples were rinsed with DI water thoroughly and dried for the next measurement. The above procedure was repeated at least 3 times for each sample.

**Evaporation Performance Measurement**

To test the performance of solar-driven water evaporation using the bifunctional membrane, the weight loss of the sample solution was measured every 30 min by an electronic balance with four-decimal precision (FR124CN, Ohaus Instrument, Shanghai, China). The weight loss of the sample solution was plotted as the function of irradiation time. In addition, an IR camera (FLIR T620, FLIR Systems, Inc., USA) was used to monitor the surface temperature of the samples during the experimental process. Before measurement, the temperature of the IR camera was calibrated using a calibrated thermocouple (Model K-H-GGF; Beijing Qiaomu Automation Technology Company, China) and the temperature uncertainty of the IR camera is ~1.0 °C.

**RESULTS AND DISCUSSIONS**

The Au NPs synthesized were visualized by TEM. The size of the particles was estimated to be ~50 nm based on a statistical calculation. After filtering through the AAO membrane, the Au NPs (0.5 mg) formed a uniform and dense packed layer on top of the AAO with layer thickness of ~1 µm (Figure 2a). As a directly contacting layer on the porous AAO substrate (with a pore size of 20 nm), the particle size of Au NPs was chose to be ~50 nm, which is large enough to avoid blocking the pores in AAO substrate during the filtration process, and also small enough to minimize the loss of TiO₂ NPs during the filtration process to deposit the TiO₂ NP layer. Compared with other assembly processes for NPs, such as evaporation assisted assembly, interferential assembly, and
spin-coating, \textsuperscript{34-36} filtration is a fast and simple approach for the generation of uniform NP films.\textsuperscript{37,38} Such a process is especially useful for the fabrication of thick NP films over a relatively large area. Another important advantage of using a filtration process in the assembly of NPs is the flexibility — both the composition and the thickness of the NP films can be easily tuned through the control of NP solutions. This flexibility also provides the possibility of tuning the microstructure and functionality of the NP films generated. In this study, to fabricate membranes with both TiO\textsubscript{2} NPs for the photocatalytic degradation function and Au NPs for the function of plasmonic enabled solar-driven evaporation, a layer of 25 nm TiO\textsubscript{2} NP film with discrete islands (Figure 2b) was formed on top of the Au NP film layer through the second filtration process after the first filtration step for the formation of Au NP film. The distinct 3-layered structure can be clearly seen with a cross-sectional SEM image under a high magnification and also EDS mapping in Figure S1 (Supporting Information). The morphology of the separated micron-sized islands of TiO\textsubscript{2} NPs helps the penetration of solar light into the underlying Au NP film. Such 3-layered membrane design provides the desired bifunctionality - photocatalysis for pollution degradation and photothermal conversion for solar-driven water evaporation, both of which contribute to the purification of the contaminated water.
Figure 2. Fabrication of bifunctional membrane. (a) Cross-sectional SEM image of 2-layer structured Au NP/AAO membrane; (b) Cross-sectional SEM image of 3-layer structured TiO$_2$ NP/Au NP/AAO membrane; (c) Preparation schematic of bifunctional membrane.

Figure 3 shows the experimental setup for the performance evaluation of the bifunctional membrane. The sample was inserted and floated on the upper part of the solution with the top surface immersed under the air-water interface. The placement of the membrane at the top portion of the solution helps localize the solar-thermal heat close to the air-water interface and thus enables efficient solar-driven evaporation process without losing heat to the bulk of the liquid underneath the membrane. The immersion of the membrane below the air-water interface facilitates the solution exchange for photo-degradation. During the measurement, the sample beaker was placed on a balance so the weight loss could be recorded in situ. The IR camera was used to measure the surface temperature of the samples.
Figure 3. Schematic setup for photocatalytic degradation and evaporation test of the bifunctional membrane under solar illumination.

The light absorbance spectra for different samples are shown in Figure 4a. The sample of AAO covered with only TiO$_2$ NPs showed good light absorbance in the wavelength range below 400 nm. Both the Au-AAO sample and the TiO$_2$-Au-AAO sample showed broadband light absorbance. This increased light absorbance should be attributed to the Au NP film underneath the TiO$_2$ NP layer. The Au NPs have surface plasmonic absorption. When the NPs form the continuous film, the plasmonic absorption delocalizes and such delocalization results in the broadening of the light absorption for the samples that contain Au NP layers.$^{39}$
Figure 4. Optical absorption and photodegradation performance of different samples: (a) Absorption spectra; (b) Photocatalytic degradation of RhB; (c) Repeated photocatalytic degradation of RhB for continuous 8 cycles.

The photocatalytic property of different membrane samples were evaluated through a liquid-phase photodegradation study of the model contaminant compound, RhB dye. As displayed in Figure 4b, under Xe light exposure with a light intensity of ~6.7 sun, the aqueous solution containing only RhB showed a very small degree of degradation. After 2h of irradiation, only ~8% of RhB was degraded. This small degree of degradation could be due to the self-degradation of RhB without any catalysts. On the other hand, the Au-AAO, TiO$_2$-AAO, TiO$_2$-Au-AAO membrane samples all showed increased photocatalytic performance. The TiO$_2$-Au-AAO membrane floating at the air-water interface showed the highest photocatalytic activity with ~ 60% of RhB degraded after 2h of light
irradiation. Without the middle Au NP layer between the TiO$_2$ NP layer and AAO layer, the photocatalytic activity decreased, and ~48% RhB was degraded after 2h for TiO$_2$-AAO membrane sample. Under the same condition, sample contains only Au NP layer on AAO showed an even lower activity in the degradation of RhB, with ~35% RhB was degraded after the same duration of light illumination. We also studied the impact of the amount of TiO$_2$ NP on photocatalytic degradation performance. As shown in Figure S2, with 2.5 fold reducing of TiO$_2$ NP amount, there are much less and smaller islands of TiO$_2$, which reduced the total surface area of the TiO$_2$ layer. Such reduced surface area actually led to the small decrease in the degradation performance (Figure S3a). The bifunctional membrane design also showed relatively good reusability. Figure 4c shows that the membrane with 0.5 mg TiO$_2$ has demonstrated stable photocatalytic degradation performance after repeating the degradation test for 8 cycles continuously.

The improvement in the photocatalytic property of the TiO$_2$-Au-AAO membrane should be attributed to the following three effects. First, the Au NP film under the TiO$_2$ film helps enhance the light absorption of the TiO$_2$ NPs through the plasmonic enhancement effect.$^{40-42}$ Such enhancement leads to the improvement in the efficiency of photoreaction of the nearby TiO$_2$ NPs. Second, the Au NP film should also facilitate electron-hole charge transfer and separation through the formation of Schottky junctions between the Au NPs and TiO$_2$ NPs, which results in the increased photocatalytic activity.$^{43}$ Third, the thermal effect caused by plasmonic photothermal conversion of the Au NP film should also play a role in the improvement of photocatalytic activity of the 3-layer composite membrane.$^{44}$

Solar-driven evaporation is an environmentally friendly and cost efficient approach that has recently attracted increased attention.$^{22-25}$ As demonstrated in our recent studies, efficient evaporation
can be achieved by taking advantage of localized heating effect of self-assembled plasmonic Au NP thin film free-floating or floating on a support at the air-water interface.\textsuperscript{26-28} Due to the confinement of the solar absorption at the top portion of the bulk liquid, the thermal energy converted from the absorbed solar light could be mostly used for the evaporation process at the evaporative surface. In this study, the localized heating effect of the Au NP film within the TiO\textsubscript{2}-Au-AAO membrane was also employed for the efficient water vapor generation. Purified water was generated after the condensation of the evaporated water. As shown in Figure 5b-e, the surface temperature of the TiO\textsubscript{2}-Au-AAO membrane was \textasciitilde 34 \degree C before Xe light illumination. After 60-min light radiation, the surface temperature reached to \textasciitilde 58 \degree C. The Au-AAO sample showed a slightly higher surface temperature of 62 \degree C than that of the TiO\textsubscript{2}-Au-AAO sample while the TiO\textsubscript{2}-AAO sample showed relatively low surface temperature of 48 \degree C after same duration of irradiation, which is attributed to the absence of the Au NP layer. The temperature rise for the TiO\textsubscript{2}-AAO samples is due to the absorption of infrared light from the Xe-lamp and also the optical-thermal conversion from the photons even within the TiO\textsubscript{2} absorption band. For the Au-AAO and TiO\textsubscript{2}-Au-AAO samples, the plasmonic photothermal effect from Au NPs converts solar energy into the thermal energy. Such conversion results in a higher interfacial temperature and also enables the higher rates of evaporation than the samples without Au NPs. As indicated in Figure 5a, without the Au NP layer, the evaporation rate for the TiO\textsubscript{2}-AAO sample is 0.24 g/cm\textsuperscript{2}/h. The Au-AAO sample and TiO\textsubscript{2}-Au-AAO sample, on the other hand, showed a much higher evaporation capability, with evaporation rates of 0.51 g/cm\textsuperscript{2}/h and 0.44 g/cm\textsuperscript{2}/h, respectively. The Au-AAO sample has a slightly higher rate of evaporation than that of TiO\textsubscript{2}-Au-AAO sample. The light absorption and scattering from the TiO\textsubscript{2} NP islands on top of the Au NP layer decreased the amount of the photons that reached the surface of the
Au NPs, and resulted in the decreased evaporation performance for TiO$_2$-Au-AAO sample.

In the 3-layered bifunctional membrane that was fabricated in this study, each layer has its desired role. The top TiO$_2$ NP layer works as the photocatalytic layer for the degradation of contamines in water. The middle Au NP layer works as the plasmonic photothermal converter for the water vapor generation and also helps enhance the photocatalytic activity through plasmonic effects. As long as the size of the Au NP is larger than the pore size of AAO membrane, the photothermal-enabled evaporation performances are close to each other for films with Au NPs of different sizes (Figure S4) due to the broad band absorption of the films. The bottom AAO membrane serves the following three functions: at the fabrication step it works as the filter for the fabrication of bifunctional membrane; later during the water purification step it serves as the supporting layer to ensure both the mechanical robustness of the composite membrane and the capillary flow of the water through the numerous pores on the membrane. Additionally, the low thermal conductivity of the AAO membrane also makes it a good thermal insulation layer to minimize the heat loss to the lower part of the solution through thermal conduction.

Figure 5. Water vapor generation by different samples. (a) Evaporation of different samples under the simulated solar light. (b-e) Surface temperature study of different samples during the testing. (b) TiO$_2$-Au-AAO sample before light illumination; (c) TiO$_2$-Au-AAO sample after 2h of light
illumination; (d) Au-AAO sample after 2h of light illumination; (e) TiO$_2$-AAO sample after 2h of light illumination.

With the three layer design, this composite membrane works as a bifunctional membrane for different degrees of water purification. After light illumination, the liquid remaining in the beaker is the partially purified water that was cleaned through the photocatalytic reaction by TiO$_2$. The evaporative function of the membrane, on the other hand, generates purified water vapor and thus purified liquid water after the condensation process. Indeed, the absorbance study showed that the liquid collected from the water vapor contains no RhB, while the solution left in the beaker after 2h of Xe light illumination contains partial purified water with ~40% of RhB remaining in the solution (Figure 6). Increasing the irradiation time would further purify the water left in the beaker to the degree that is needed for different applications.

**Figure 6.** Water purification by bifunctional membrane: (a) absorption before and after purification for 2h; (b) optical images of the water in cuvettes before and after purification for 2h.
CONCLUSION

In summary, a 3-layer bifunctional composite membrane with photocatalytic function and photo-thermal driven evaporation function was designed and fabricated for efficient water purification using solar light. Previously, the loss of energy efficiency in the photocatalytic water purification has been the result of two processes: 1) the absorption process with relative narrow absorption band of photocatalysts; and 2) the photo-thermal conversion with the absorbed photons. The integration of the photo-thermal driven evaporation function with the photocatalytical function helps expand the range of useful solar light for the clean water generation, and also takes advantage of the heat generated by the catalysts for further efficiency enhancement. Such multi-layered membrane enables the generation of both pure and partial purified water in a single step. Pure water was generated through the photo-thermal evaporation process and subsequent condensation, and partially purified water was the result of the photocatalytic degradation process. Compared to the membrane with only a single functionality, the bifunctional design provides a new approach for water purification, and also opens up an alternative strategy to maximize solar energy conversion and utilization. This reported membrane design could be further improved, for example by replacing the Au NPs with other low cost photothermal converters, such as carbon NPs, and by utilizing flexible porous substrates instead of the rigid ceramic AAO substrate, to explore practical applications for pure water generation in large scale in the future.

ASSOCIATED CONTENT

Supporting Information.

Energy dispersive spectroscopy (EDS) mapping of the TiO$_2$-Au-AAO membrane; SEMs and
performance of membranes with TiO$_2$ NP layer of different thicknesses; evaporation rate of membranes with Au NP (50 nm and 100 nm in diameter) layer of different thicknesses. This material is available free of charge via the Internet at http://pubs.acs.org.”

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Notes

The authors declare no competing financial interest.

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Bioinspired membrane that demonstrates the bifunctional performance of water purification: 1) the generation of purified water through photocatalytical degradation of contaminants; 2) the generation of clean condensed water through solar-driven water evaporation process. Such bifunctional membrane offers an alternative approach in generating decontaminated water, and also potentially improves the efficiency in solar energy usage.