Green-synthesizing Ag nanoparticles by watermelon peel extract and their application in solar-driven interfacial evaporation for seawater desalination

Hongxia Cao\textsuperscript{1,2,3,4}, Tianlei Cui\textsuperscript{3}, Wenyuan Wang\textsuperscript{3}, Shilei Li\textsuperscript{3}, Xueyun Tang\textsuperscript{3}, Hongyan Wang\textsuperscript{1,3} and Guang Zhu\textsuperscript{1,4}

\textsuperscript{1} Key Laboratory of Spin Electron and Nanomaterials of Anhui Higher Education Institutes, Suzhou University, Suzhou 234000, People’s Republic of China
\textsuperscript{2} Jiangsu Province Engineering Laboratory of High Efficient Energy Storage Technology and Equipments, China University of Mining and Technology, Xuzhou 221116, People’s Republic of China
\textsuperscript{3} School of Chemistry and Chemical Engineering, Suzhou University, Suzhou 234000, People’s Republic of China
\textsuperscript{4} Authors to whom any correspondence should be addressed. E-mail: hxcao@ahszu.edu.cn and guangzhu@ahszu.edu.cn

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Abstract

Ag nanoparticles displayed a more attractive application in solar-driven interfacial evaporation for seawater desalination, but their synthesis process was expensive and detrimental to the environment due to the consumption of chemicals. In this work, we green-synthesized Ag nanoparticles using watermelon peel extract as reducing agent, while the membrane with Ag nanoparticles was prepared for solar-driven interfacial evaporation. Moreover, the evaporation performance of the membranes with different Ag nanoparticles loadings was investigated, as well as the reusability and desalination application was also explored. The experimental results showed that the membrane with Ag nanoparticles loading of 0.05 g exhibited superior evaporation performance, corresponding to the evaporation rate of 1.37 kg m\textsuperscript{-2}h\textsuperscript{-1} and the evaporation efficiency of 89.3\%, respectively. In addition, the membrane with Ag nanoparticles possessed an excellent recyclability and concentration of ions in the desalinated seawater reached the potable water standard. Our research demonstrated that Ag nanoparticles green-synthesized by watermelon peel extract was an ideal candidate applied in solar-driven interfacial evaporation for seawater desalination.

1. Introduction

Global water resources, especially freshly potable water, is confronting a serious water shortage crisis due to the ever-increasing population and ever-worsening environment stemming from industrial development\textsuperscript{[1–3]}. Although there is abundant seawater in the Earth, it cannot be directly utilized as drinkable water due to its high salinity. Intentionally, sustainable seawater desalination is considered as a chief means and valid approach to gain fresh water in many regions, particularly water-stressed area. At present, commercial desalination technologies mainly involve two major categories, one of which is membrane-based designs like reverse electrodialysis and reverse osmosis\textsuperscript{[4, 5]}, and the other of which is thermal-based designs such as vapor compression distillation and multi-stage flashing\textsuperscript{[6]}. Nevertheless, high energy consumption combined with costly equipment make these desalination technologies unrealistic in underdeveloped region. Alternatively, solar steam generation is low-cost and easy to achieve small-scale production with free renewable solar energy, which is regarded as a promising solution for freshwater supply in these economically impoverished areas and remote areas\textsuperscript{[7, 8]}. For solar steam generation, water evaporation really occurs at the water–air interface. However, traditional solar evaporation system generates vapor by warming the entire body of water, which results in lower photo-thermal conversion efficiency\textsuperscript{[9]}. Therefore, it is still extremely challenging to realize efficient absorption and conversion of solar energy.
Recently, the floating structure has attracted sweeping attention from researchers worldwide due to its effective thermal aggregation properties at the evaporation interface [10–12]. Miao et al [13] developed a simplified solar evaporation system using floating structure with carbon nanotubes membrane as a light absorber and aerogel blanket as a thermally insulating layer, which attained a high evaporation efficiency of 84.6% at a light intensity of 1 kW m$^{-2}$. In this case, the most important part of solar evaporation system is the photo-thermal conversion material, which is directly related to harvesting solar energy, affecting the evaporation performance [14–17]. At present, metal (especially for Au, Ag) nanoparticles were reported as better photo-thermal conversion materials due to localized surface plasmon resonance (LSPR), which induced collective oscillation of charges in the metal nanoparticles to produce hot electrons [18–21]. For example, Chen et al [22] employed a seed mediated method to synthesize Au nanoparticles with different diameters and the results demonstrated that an enhanced light absorption at a low mass fraction of Au nanoparticles relative to working fluid was ascribed to LSPR. Compared to Au, Ag displayed a more attractive appeal in solar thermal energy owing to its lower price with comparable photo-thermal conversion performance [23–26]. As reported by Chen et al [27], Ag nanoparticles were fabricated by an effective seeded growth strategy with limited spaces, which promoted absorption of sunlight from the near-infrared region to the visible region. Unfortunately, the synthesis of Ag nanoparticles was mainly achieved by chemical reduction methods, which were more expensive and detrimental to the environment due to the consumption of chemicals, such as sodium borohydride, sodium citrate and so on [28]. Therefore, it still has a significant challenge to attain Ag nanoparticles for photo-thermal conversion using an energy-saving and eco-friendly feasible alternative.

In this work, we green-synthesized Ag nanoparticles using watermelon peel extract as reducing agent, and prepared the membrane with Ag nanoparticles for solar-driven interfacial evaporation. Moreover, Ag nanoparticles were analyzed by x-ray diffraction (XRD) and transmission electron microscope (TEM), and the membrane with Ag nanoparticles was characterized using scanning electron microscope (SEM) and water contact angle, respectively. In addition, the evaporation performance of the membranes with various Ag nanoparticles loadings was explored, as well as the reusability and desalination application was also tested.

2. Experimental

2.1. Materials
Shanghai 3F New Materials Co., Ltd and Hangzhou Special paper Industry Co., Ltd commercially supplied polyvinylidene fluoride (PVDF) and qualitative filter paper (GB/T 1914–2007), respectively. Silver nitrate (AgNO$_3$), sodium chloride (NaCl), sodium hydroxide (NaOH), N, N-Dimethylformamide (DMF), polyvinyl pyrrolidone (K30) and sodium dodecyl sulfate (SDS) were analytical reagents, which were obtained from Sinopharm Chemical Reagent Co., Ltd Watermelon peel was collected from the market located at Suzhou, Anhui, China. Deionized (DI) water was homemade.

2.2. Green synthesis of Ag nanoparticles
Watermelon peel washed with DI water was cut into small pieces and then processed into a water-soluble extract using a wall breaking machine. After 10 min centrifugation at 10000 rpm, the supernatant was stored at 4 °C. Ag nanoparticles were green-synthesized using the supernatant as reducing agent, which can be regarded as an environmentally friendly preparation technique. Typically, 5 mmol AgNO$_3$ was dissolved into 10 ml DI water, followed by adding 4 g K30 to form a uniform solution. Subsequently, 100 ml supernatant was poured into the above solution under stirring and then its pH value was adjusted to 10.0 using 0.5 M NaOH solution. The resulting solution was stirred continuously until it turned black, indicating that the chemical reaction of Ag$^+$ to Ag$^0$ was completely completed. Ultimately, Ag nanoparticles were acquired by centrifugation and vacuum drying.

2.3. Preparation of membrane
As seen from figure 1, the membrane with Ag nanoparticles was prepared as follows. Firstly, 0.01 g Ag nanoparticles were dispersed into 50 ml DI water under stirring, which was accompanied by a follow-up ultrasound for 30 min to obtain a suspension. After suction filtration, a thin Ag nanoparticles film formed on qualitative filter paper, which was dried at 60 °C under vacuum for 30 min. PVDF was dissolved into DMF according to a ratio of 1:10 at 60 °C under stirring for 30 min to obtain a PVDF solution. Then, 1 ml PVDF solution was dripped onto Ag nanoparticles film at 200 r min$^{-1}$ for 15 s and 500 r min$^{-1}$ for 20 s to gain a membrane with Ag nanoparticles. After drying under vacuum for 30 min to remove overfull DMF, the membrane with Ag nanoparticles was cut into a disc with a predetermined diameter of 35 mm, which was named as membrane I. Additionally, the membranes with Ag nanoparticles of 0.03, 0.05 and 0.07 g were fabricated using the identical experimental procedure, which were designated as membrane II, membrane III.
and membrane IV, respectively. As a comparison, the membrane without Ag nanoparticles was selected as the control sample, which was labeled as membrane 0.

2.4. Characterization
XRD pattern of Ag nanoparticles in the range of 10° to 80° was measured by SmartLab3KW diffractometer (Cu Kα, 40 kV, 40 mA). TEM images of Ag nanoparticles were employed to explore their morphologies and recorded on a FEI TECNAI G2F20 S-TWIN instrument. SEM images of membranes were attained on the Quanta 250 equipment of FEI Company. Water contact angle was achieved by a JC2000C1 contact angle goniometer from Shanghai Zhongchen Digital Technic Apparatus Co., Ltd. An infrared thermal imager was applied to measure the surface temperature of membrane (TI100, Fluke, USA) and light intensity was acquired using a CEL-NP2000 optical power meter. Concentration of ions in seawater was monitored by ICP obtained on the NWR213–7900ICP-MS instrument.

2.5. Evaporation experiment
Figure 2 described the schematic illustration of solar-driven interfacial evaporation system. Evaporation performance was evaluated in a self-made cylindrical container (inner diameter: 36 mm, height: 85 mm), which was filled in salt water with 3.5 wt% NaCl. Simulated solar spectrum was acquired using a filtered AM1.5 light provided by a CEL-HXF300 xenon lamp. The membrane was utilized to harvest solar radiation, as well as the paper belt and XPS extruded board were used as water transmission pipeline and thermal insulator for heat localization, respectively. This experiment was performed at a light intensity of 1 kW m⁻² for 30 min illumination and the measurement of mass change was conducted on a JJ224BF electronic balance. The initial temperature of salt water was kept at 25 °C and the surrounding temperature was invariably maintained at 25 °C.

Evaporation rate, described as the amount of steam generated per unit area per unit time, was calculated based on previously published literature [29]:

![Figure 1. Schematic of fabrication process of membrane with Ag nanoparticles.](image1)

![Figure 2. Schematic illustration of solar-driven interfacial evaporation system.](image2)
in which, \( R_e \) stands for the evaporation rate (kg m\(^{-2}\)h\(^{-1}\)), \( \Delta m \), \( A \) and \( t \) represent the mass loss (kg), irradiation area (m\(^2\)) and irradiation time (h), respectively. Additionally, evaporation efficiency, which was also regarded as a crucial indicator of evaporation performance, was defined as the following equation [29]:

\[
\eta = \frac{R_e \times h_{LV}}{3600q}
\]

where \( \eta \) and \( q \) denote the evaporation efficiency (%) and light intensity (kW m\(^{-2}\)), respectively. Importantly, \( h_{LV} \) mainly related to the sensible heat due to temperature increase of water and the latent heat owing to phase change of water to steam, which was expressed as follows [29]:

\[
h_{LV} = C\Delta T + \Delta h
\]

where \( C \) was the water specific heat capacity (4.18 kJ kg\(^{-1}\) K\(^{-1}\)), \( \Delta T \) the temperature rise (K), \( \Delta h \) the evaporation enthalpy (2257 kJ kg\(^{-1}\)).

3. Results and discussion

3.1. Characteristics

XRD pattern of Ag nanoparticles in the range of 10°–80° was illustrated in figure 3. Four strong diffraction peaks were able to be distinctly observed at \( 2\theta \) of 38.3°, 44.5°, 64.6° and 77.6°, which were respectively associated with (111), (200), (220) and (311) reflection plane of face centered cubic Ag [30], indicating that Ag nanoparticles were successful synthesized using watermelon peel extract based on a green reduction scheme. Noting that, the faint peaks at \( 2\theta \) of 27.7°, 32.5° and 46.4° were also shown in this XRD curve, corresponding to AgCl nanoparticles as described in [31]. The reason was that, during Ag nanoparticles green synthesis process, a very small amount of AgCl compounds may be yielded because Ag\(^+\) can be bonded to a trace amount of Cl\(^-\) from watermelon peel extract.

Figures 4(a) and (b) exhibited TEM images of Ag nanoparticles at different magnification. Obviously, the morphology of Ag nanoparticles was approximately spherical. Moreover, no significant aggregation was observed between nanoparticles owing to the role of capping agents including K30 and organic components derived from watermelon peel extract, reflecting the superior stability of Ag nanoparticles. 100 nanoparticles in TEM image were randomly selected to calculate particle size. In can be found from figure 4(c) that the diameter of Ag nanoparticles varied from 8 to 19 nm and their average value was about 13 nm.

SEM images of qualitative filter paper and membrane with Ag nanoparticles at different scales were described in figure 5. As seen from figure 5(a), qualitative filter paper with layered structure was made up of a plurality of ribbon fibers stacked layer by layer. The layered structure was characterized by a porosity and large specific surface area, which was beneficial to the escape of generated steam during desalination. According to the enlarged SEM image in figure 5(b), the wrinkle morphology was clearly observed on the surface of single fiber, which was advantageous for Ag nanoparticles loading. After incorporation of Ag nanoparticles, as shown in
figure 5(c), the membrane still retained the porous structure. Moreover, Ag species was evenly dispersed on the membrane, which was conducive to promoting light absorption and consequently elevating water evaporation. From figure 5(d), Ag nanoparticles were tightly attached to the membrane, suggesting superior stability due to intimate interaction between them. Additionally, the infiltration of droplet on membrane was probed by dynamic contact angle, as plotted in figures 5(e) and (f), water contact angle of 69.7° was initially detected on membrane, implying that the membrane was hydrophilic. After standing for 2 s, the water contact angle was changed from 69.7° to 54.5°, which reflected that water diffused into the porous structure of membrane due to the capillary force. Simultaneously, the capillary force was also able to be employed to drive water into the evaporation region for generating steam.

3.2. Photo-thermal conversion performance of membrane
Figure 6 demonstrated the change of surface temperature of membranes over time. Noting that, a rapid temperature increase was observed at the initial stage, and then the temperature remained unchanged over time, indicating that heat equilibrium was achieved between solar absorption and thermal dissipation. Membrane 0 exhibited the lowest average temperature rise of 36.2 °C while membranes I-IV possessed higher average temperature rise, suggesting that the addition of Ag species upgraded the ability of light absorption due to LSPR of Ag nanoparticles. Additionally, Ag nanoparticles loading played a crucial role in temperature change. The temperature increased rapidly with increasing Ag nanoparticles loading below 0.05 g, corresponding to a mean temperature of 85.0 °C with 0.05 g Ag nanoparticles loading, and only slight temperature change was observed above 0.05 g Ag nanoparticles loading. This phenomenon revealed that the proper amount of Ag nanoparticles
loading was conducive to well dispersion of Ag nanoparticles on the membrane, thereby improving light absorption performance to yield more localized heat for steam generation.

3.3. Solar-driven interfacial evaporation

Ag nanoparticles loading played a critical role in solar-driven interfacial evaporation. As shown in figure 7(a), mass loss was merely 0.26 g when the membrane 0 was exposed to sunlight for 30 min. Under the same condition, a remarkable improvement in mass loss was noted using the membrane with Ag nanoparticles. Specifically, mass loss increased significantly with elevating Ag nanoparticles loading from 0.01 to 0.05 g. This was because more Ag nanoparticles dispersing on the membrane can efficiently absorb incident light and converted it to heat for steam generation, thus enhancing evaporation performance. Further increase in Ag nanoparticles loading only caused a slight change of mass loss, which responded to 0.68 g at Ag nanoparticles loading of 0.05 g and 0.69 g at Ag nanoparticles loading of 0.07 g, respectively. This was explained by Ag nanoparticles aggregation due to the higher loading, which was detrimental to light absorption. The above analysis demonstrated that the optimum Ag nanoparticles loading was 0.05 g. Further, similar trends were observed in evaporation rate and evaporation efficiency according to figure 7(b). Obviously, evaporation rate and evaporation efficiency of the membrane III were 1.37 kg m$^{-2}$h$^{-1}$ and 89.3%, considerably better than
0.574 kg m\(^{-2}\) h\(^{-1}\) and 36.4% of the membrane 0. This also signified that the suitable Ag nanoparticles loading had a decisive influence on evaporation performance.

Temperature-time curve of surface temperature of membranes with different Ag nanoparticles loadings was plotted in figure 8(a). After 30 min solar evaporation, a surface temperature of nearly 30 °C was clearly visible on the membrane 0. Moreover, incorporating Ag nanoparticles induced that the surface temperature was rapidly raised to about 40 °C due to LSPR of Ag nanoparticles, thereby promoting solar steam generation. As Ag nanoparticles loading increased from 0.01 to 0.07 g, the surface temperature gradually elevated and stabilized at around 45 °C. The surface temperature of the membranes after 30 min illumination was further identified by the images in figure 8(b). Apparently, the surface temperature of membrane increased from 39.86 to 44.66 °C with a rise of Ag nanoparticles loading, corresponding to a relatively stable value of about 44.6 °C at Ag nanoparticles loading of 0.05 g, which was higher than 29.8 °C of the membrane 0. These analysis results were consistent with evaporation rate and evaporation efficiency discussed above.

### 3.4. Reusability and desalination application

Stability testing was very essential for seeking top-quality candidate in long-term seawater desalination. Therefore, a 10-cycle lifetime test was performed on the membrane III at 1 kW m\(^{-2}\) light intensity for seawater desalination and the results was shown in figure 9. Evaporation rate and evaporation efficiency of the membrane III maintained practically constant during the entire cycle test, which responded to 1.37 kg m\(^{-2}\) h\(^{-1}\) and 89.3%, respectively, suggesting an excellent recyclability of the membrane with Ag nanoparticles in seawater desalination. In addition, for further exploring the practical application prospect of the membrane with Ag nanoparticles,
nanoparticles in seawater desalination, concentration of ions in seawater before and after desalination was evaluated by ICP, as described in figure 10. There were mainly Na$^+$, Mg$^{2+}$, K$^+$, and Ca$^{2+}$ in seawater, and their concentrations were 1371.20, 453.65, 500.70 and 38.25 mg l$^{-1}$ before desalination. Nevertheless, after desalination, the corresponding ion concentrations separately dropped to 2.65, 0.02, 0.32 and 0.01 mg l$^{-1}$, which met the potable water standards [32]. The above analysis further illustrated that the green-synthesized Ag nanoparticles possessed broad application prospects in terms of seawater desalination.

4. Conclusions

In this work, Ag nanoparticles with small particle size were successful synthesized by watermelon peel extract through a green reduction scheme, which were applied in solar-driven interfacial evaporation for seawater desalination. The experimental results showed that Ag nanoparticles were evenly dispersed on the membrane and the addition of Ag species upgraded the ability of light absorption due to LSPR. Moreover, the suitable Ag
nanoparticles loading had a decisive influence on evaporation performance, and the membrane with Ag nanoparticles loading of 0.05 g exhibited superior evaporation performance, corresponding to the evaporation rate of 1.37 kg m\(^{-2}\)h\(^{-1}\) and the evaporation efficiency of 89.3%, respectively. In addition, the membrane with Ag nanoparticles possessed an excellent recyclability and concentration of ions in desalinated seawater reached the potable water standard, which demonstrated that Ag nanoparticles green-synthesized by watermelon peel extract is a promising choice applied in solar-driven interfacial evaporation for seawater desalination.

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ORCID iDs

Hongxia Cao @ https://orcid.org/0000-0003-2484-9378

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