Iron Based Chitin Composite Films for Efficient Solar Seawater Desalination

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Abstract: Seawater desalination provides a convenient method for the sustainable production of fresh water. However, the preparation of low-cost, high-efficiency solar absorbers remains a huge challenge. To this end, our research group designed and produced a cheap absorber—a membrane made of natural polymer chitin with black FeS and Fe3O4, respectively. Due to the hierarchical pore structure, excellent photothermal performance and good hydrophilicity of the film, their water evaporation rates reached 1.47 kg/m²/h and 1.55 kg/m²/h under one sunlight, respectively. Under about 10 suns, the highest desalination efficiency of FeS/chitin and Fe3O4/chitin are 90% and 74%, respectively, and their salinities are also in line with the World Health Organization drinking water standards. These results indicate the potential of chitin-based nanomaterials as high-efficiency solar absorbers to produce fresh water.

Keywords: seawater desalination; nanoparticles; chitin; FeS; Fe3O4

1. Introduction

Water is the source of life. With the rapid development of society and the overuse of freshwater resources, many countries and regions are facing the problem of freshwater shortage. More than 70% of the earth’s surface is ocean; therefore, the extraction of fresh water from the ocean is a valuable technological method [1–3]. Traditional seawater desalination technology, however, has the disadvantages of high energy consumption, serious pollution and low efficiency. For example, the traditional desalination method mainly includes distillation, reverse osmosis membrane and multi-stage flash method. These methods are either at the cost of energy consumption or demanding for pretreatment of seawater. The most abundant energy source of solar energy will be a substitute for traditional fossil fuels and a low-carbon energy source, which will not aggravate global warming like fossil fuels. Therefore, solar seawater desalination technology came into being, and solar desalination is expected to solve the freshwater shortage crisis [4–6]. The membrane distillation technology using solar energy has the advantages of green, environmental protection and no non-renewable energy. The key to the high efficiency of solar seawater desalination lies in the selection of photothermal materials.

In recent years, the use of photothermal materials that can absorb solar radiation and convert solar energy into thermal energy has attracted wide attention [7–10]. Based on this, many scientific researchers are trying to develop a new type of light-to-heat conversion technology; that is, solar membrane distillation technology. It has a broad application prospect in solving the global shortage of fresh water resources. Recently, Zhang et al. prepared multifunctional textiles composed of Ag3PO4-rGO nanocomposite coated on a cotton fabric substrate with the water evaporation rate of 1.31 kg/m²/h under
sunlight [11]. Wang et al. reported a general method for constructing photothermal layers on different substrates, which enhanced the interfacial water evaporation to 1.55 kg/m$^2$/h through polypyrrole (PPy) polymerization and silver nanoparticle (Ag NPs) deposition [12]. Jun et al. prepared a polydopamine/hydroxyapatite based nanowire bilayer, showing a rate of 0.89 kg/m$^2$/h [13]. Peng et al. designed an independent MOF based membrane with excellent photothermal properties. Under one sun, its evaporation rate reached 1.53 kg/m$^2$/h [14]. However, the application of iron-based nanoparticles loaded on natural polymer chitin for solar-driven interfacial water evaporation is rarely reported.

FeS and Fe$_3$O$_4$ nanoparticles are widely used because of their advantages of good infrared photothermal effect, low toxicity, biocompatibility, low cost, easy processing and hydrophilicity [15–17]. Chitin is a common natural polymer material, which has been applied in practice because of its wide source, environmental benignness, excellent chemical stability, biocompatibility, and low toxicity [18–20]. Chitin can be used as materials in the adsorbent, antibacterial, catalytic technology, Fenton reaction and other aspects of extensive research. Because of its good biocompatibility and being non-toxic, it can also serve as a biological material. In this work, we conducted solar desalination experiments by loading the well-absorbed FeS and Fe$_3$O$_4$ onto chitin, respectively. The composite films showed high desalination efficiency. Under about 10 suns, the highest desalination efficiency of FeS/chitin and Fe$_3$O$_4$/chitin are 90% and 74%, and the water evaporation rates reached 1.47 kg/m$^2$/h and 1.55 kg/m$^2$/h under one sunlight, respectively, and their salinities are also in line with the World Health Organization drinking water standards. The preparation method is easy to operate and can be used as a universal and scalable method to construct different photothermal materials on chitin substrates, demonstrating the potential application in the field of photothermal evaporation.

2. Experimental Part
2.1. Experimental Materials

Sea salt (Shanghai Guangyu Biotechnology Co., Ltd., Shanghai, China), chitin (Golden-Shell Pharmaceutical Co. Ltd., Shenzhen, China), FeS, Fe$_3$O$_4$, sodium hydroxide, sulfuric acid, urea, are analytical pure.

2.2. Experimental Process
2.2.1. Chitin Purification

100 g chitin was dispersed in 400 g NaOH (5 wt%) solution, stirred for 10 h, washed with distilled water to neutral, and filtered. Afterwards, the product was stirred in HCl (7 wt%) aqueous solution for 36 h to remove excess protein, washed with distilled water to neutral, and filtered. Then the collected chitin was dispersed in 400 g NaOH (5 wt%) aqueous solution, stirred for 10 h, washed with distilled water to neutral, and filtered. The sample was then bleached in sodium acetate (0.3 M) buffer solution containing sodium chlorite (1.7 wt%) at 80 °C for 6 h to remove the residual pigment, washed with distilled water to neutral, and filtered. After being dried, the purified chitin was stored in a dryer.

2.2.2. Dissolution of Chitin and Its Composites

7 g of the purified chitin was dispersed in 11 wt% NaOH- 4 wt% urea- 85 wt% H$_2$O solutions and stirred for 15 min. FeS and Fe$_3$O$_4$ of different proportions were then added to the dispersion, respectively, and stirred for 1 h. The dispersion was then frozen in the refrigerator overnight, stirred and thawed at room temperature. After repeating the freeze-thaw process twice, the black FeS/chitin and Fe$_3$O$_4$/chitin aqueous solution were obtained.

2.2.3. Preparation of Fe-Based Chitin Composite Films

A 0.5 cm thick liquid film was obtained by casting. FeS/chitin and Fe$_3$O$_4$/chitin composite films were obtained by regenerating the films in H$_2$SO$_4$ (5 wt%) solution at 0 °C. Then the composite films were dialyzed in distilled water for 3 d to remove the excess
alkali and urea. After being stripped and dried, the bright FeS/chitin and Fe$_3$O$_4$/chitin composite films were obtained. By changing the concentration of FeS and Fe$_3$O$_4$, it is proved that a Fe base mass of 30 wt% can achieve the best desalination effect.

2.2.4. Artificial Seawater Preparation

A certain amount of sea salt was added to the container containing ultrapure water, then ultrasonically stirred. The appropriate amount of solution with 3.5 wt% was then obtained using a salinity meter.

2.2.5. Desalination Performance of FeS/Chitin and Fe$_3$O$_4$/Chitin Films

The composite film was put onto a tripod and moved into a solar reactor connected with a condensing equipment. Then a certain amount of seawater was added. After irradiated under 300 W xenon light, the tripod was weighed and the desalination efficiency was calculated.

2.3. Measurement

The morphology was analyzed via a Sigma Zeiss Field sweep surface electron microscope (FESEM) and an energy dispersive X ray spectrometer (EDS) was used to analyze the sample composition. High-resolution transmission electron microscopy (HRTEM) images were obtained by JEOL JEM-2100F (UHR) field emission TEM. X diffraction patterns (XRD) were recorded by a D8 Advance powder X diffractometer (Bruker, Karlsruhe, Germany). The surface area and N$_2$ adsorption-desorption curves were obtained by N$_2$ analyzer. The particle size distribution was obtained using a BJH model. Infrared spectrum (IR) was obtained on Nicolet iS5 fourier IR instruments after compressed by KBr. UV-vis diffuse reflectance (DRS) was obtained through the UV-vis Spectrometer (Perkin Elmer, Waltham, MA, USA, Lambda 850, BaSO$_4$ as a reference) in the range of 200–1400 nm. The X-photoelectron spectroscopy (XPS) data was obtained by X ray photoelectron spectrometer (Thermo Escalab 250XI, Waltham, MA, USA). Thermal imaging images and real-time temperature monitoring were measured by infrared thermal imager (Sweden, FLIR, A300).

3. Results and Discussion

3.1. Morphology and Structure of Materials

Figure 1 shows SEM images of FeS/chitin and Fe$_3$O$_4$/chitin films. As shown in Figure 1, FeS and Fe$_3$O$_4$ nanoparticles were grown on the chitin nanofibers. Seen from Figure 1a,b, the structure of chitin didn’t change in the composite films. The average size of the FeS is between 10–30 nm. Figure 1c,d displayed that Fe$_3$O$_4$ is hexahedron with the average size of about 100 nm. Nanoparticles are attached to the surface or pores of chitin fibers. Therefore, a composite membrane composed of chitin nanofibers and nanoparticles was successfully constructed using a simple green method. The synthesis of nanoparticles on nanofiber chitin is an important step to promote its wide application. Figure 1e,f are scanned images of pure chitin.

3.2. Fixation and Size Distribution of Nanoparticles on Chitin

Figure 2 shows the nitrogen adsorption isotherms and pore size distribution of chitin, FeS/chitin and Fe$_3$O$_4$/chitin films. According to the nitrogen adsorption isotherm and pore size distribution data, the area, pore volume and pore size data of chitin, FeS/chitin and Fe$_3$O$_4$/chitin composite films are shown in Table 1. Chitin still maintains its nanoporous structure after the deposition of FeS and Fe$_3$O$_4$. The main pore sizes of the samples are in the range of 10–150 nm, while the reduction of chitin surface area and pore volume may be due to the aggregation of nanoparticles.
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Figure 2 shows the nitrogen adsorption isotherms and pore size distribution of chitin, FeS/chitin and Fe₃O₄/chitin films. According to the nitrogen adsorption isotherm and pore size distribution data, the area, pore volume and pore size data of chitin, FeS/chitin and Fe₃O₄/chitin composite films are shown in Table 1. Chitin still maintains its nanoporous structure after the deposition of FeS and Fe₃O₄. The main pore sizes of the samples are in the range of 10–150 nm, while the reduction of chitin surface area and pore volume may be due to the aggregation of nanoparticles.

Table 1. Surface properties of Chitin and Fe/Composite membrane.

| Sample           | Surface Area (m²·g⁻¹) | Pore Volume (cm³·g⁻¹) | Pore Size (nm) |
|------------------|------------------------|------------------------|----------------|
| Chitin           | 149.860                | 1.305                  | 17.388         |
| FeS/Chitin       | 120.560                | 0.954                  | 17.408         |
| Fe₃O₄/Chitin     | 80.375                 | 0.668                  | 17.442         |

TEM Figure (Figure 3) showed that the size of the nanoparticles is consistent with the SEM, and also verified the successful loading of nanoparticles on chitin. Figure 3a,b showed that the lattice fringe spacing was 0.256 nm and corresponded to the (210) crystal plane of FeS. Known from Figure 3e,f, the crystal plane spacing of 0.2916 nm corresponded
to (422) crystal plane of Fe₃O₄, proving its hexagonal lattice. Meanwhile, the corresponding mapping diagram also confirmed the existence of FeS and Fe₃O₄.

XRD data can also verified the FeS, Fe₃O₄ fixation on the chitin matrix. The XRD spectra of the composite films and chitin were shown in Figure 4a. There were five peaks at 9.3°, 12.7°, 20.9°, 23.6°, 26.4° in the XRD spectra of chitin, corresponding to (020), (021), (120), (130) and (013) planes, confirming the typical antiparallel structural of chitin [21,22]. In addition to the five characteristic peaks of the chitin peak, the composite film all showed their own characteristic peaks. For FeS/chitin film, four additional peaks at 30.2°, 33.9°, 43.5° and 53.4° corresponded to (110), (112), (114), and (300) planes of FeS, indicating the regular hexagonal lattice of FeS nanoparticles [23]. Besides chitin and FeS, there are no other peaks in the diffraction pattern, indicating the successful loading of FeS on chitin. Fe₃O₄/chitin film exhibited characteristic peaks of Fe₃O₄ at 30.1°, 35.4°, 43.1°, 53.4°, 56.9° and 62.5°, corresponding to the (220), (311), (400), (422), (511) and (440) crystal planes, implying the hexagonal crystal structure of Fe₃O₄ (JCPDS 19-0629) [24]. Meanwhile, after loading the nanoparticles, the crystal structure of chitin in the composite remains unchanged. The UV-Vis-NIR DRS of chitin and composite films were shown in Figure 4b. As seen from the diagram, chitin had absorbance only in the ultraviolet region, while FeS/chitin and Fe₃O₄/chitin showed strong absorption throughout the spectrum, the absorption intensity of FeS/chitin and Fe₃O₄/chitin in the visible-infrared region is obviously improved, indicating that the addition of nanoparticles was beneficial to improve the efficiency of solar light absorption.

Figure 3. (a) TEM and (b) HRTEM of FeS/chitin composite film; (c,d) FeS/chitin composite film mapping; (e) TEM and (f) HRTEM of Fe₃O₄/chitin composite film; (g,h) Fe₃O₄/chitin composite film mapping.
3.3. Interaction between FeS, Fe$_3$O$_4$ and Chitin

As mentioned above, FeS and Fe$_3$O$_4$ nanoparticles were firmly immobilized on chitin nanofibers. FT-IR and XPS were conducted to characterize the possible interactions between FeS, Fe$_3$O$_4$ and chitin. The IR spectra of samples were shown in Figure 4c. The strong absorption peak at 3430 cm$^{-1}$ corresponded to the hydrogen bond and hydroxyl stretching vibration of chitin matrix [25,26]. Two typical peaks at 1670 cm$^{-1}$ and 1580 cm$^{-1}$ corresponded to the acetamide type I and II stretching vibration, respectively [27]. The absorption peak at 1070 cm$^{-1}$ corresponded to C-O-C. Compared with FeS/chitin and Fe$_3$O$_4$/chitin, chitin showed decreased intensity for hydroxyl groups, acetamide bond and O-C-O, indicating that FeS and Fe$_3$O$_4$ were successfully loaded on chitin.

Figure 5 showed the XPS spectra of chitin, FeS/chitin and Fe$_3$O$_4$/chitin films. There are Fe and S in addition to the C, N, O the spectra for FeS/chitin. Figure 5a clearly show strong Fe 2p 3/2 and Fe 2p 1/2 peaks at 711.2 eV and 724.5 eV, respectively. The weak signal between the two major peaks may be due to the presence of iron oxide(Fe$_2$O$_3$). The peak at 164.0 eV in Figure 5b is attributed to S$^{2-}$ according to the standard spectrum [28,29]. Because S$^{2-}$ can be oxidized in the air, the peak at 168.5 eV assigned to the SO$_3^{2-}$ were also observed [30]. The Fe2p XPS spectra (Fe$_3$O$_4$ nanoparticles) show two prominent peaks. The two peaks correspond to the Fe 2p3/2 and the Fe 2p1/2, respectively. Two peaks at 710.2 eV and 724.5 eV corresponded to Fe$^{2+}$ and the peaks at 712.0 eV and 725.1 eV belonged to Fe$^{3+}$, which is consistent with the spectra of the Fe$_3$O$_4$ reported in the literature [31]. There were two peaks at 714.9 eV and 727.5 eV, indicating the existence of small amount of Fe$_2$O$_3$ [31,32].
The C 1s spectra at 284.64 eV, 286.15 eV and 287.76 eV corresponded to C-C/C-H, C=O/C-N and C=O/O-C-O groups, respectively (Figure 5d) [33,34]. After loading FeS, the corresponding binding energies were 284.57 eV, 286.17 eV and 287.91 eV, respectively, while after the load of Fe3O4, the corresponding binding energies were 284.62 eV, 286.22 eV and 287.87 eV, indicating that there is no interaction between C and FeS nor Fe3O4. Three peaks of O 1s are located at 531.11 eV, 532.50 eV and 535.25 eV, corresponding to N=O=C, C-O-C and O-C-O, respectively (Figure 5f) [35–37]. After loading FeS, the corresponding binding energies were reduced to 531.07 eV, 532.57 eV and 535.15 eV. When Fe3O4 was loaded, the binding energies were 529.50 eV, 532.64 eV, 535.22 eV, indicating the interaction between O and FeS or Fe3O4. The N1s peak at 399.55 eV corresponded to acetamide groups [38], which increased to 399.69 eV after loading FeS and increased to 399.74 eV after the load of Fe3O4, indicating that there is also interaction between N and FeS or Fe3O4.

3.4. Desalination Performance of Composite Films

XPS results also confirmed the successful loading of FeS, Fe3O4 on the chitin. FT-IR analysis results are in consistent with XPS results, indicating that FeS, Fe3O4 interact with N (N=C=O) and O (O=C-N) through coordination bonds. It is highly probable that FeS, Fe3O4 were immobilized on chitin nanofibers by coordination with acetamide groups. Hence, the firm immobilization of nanoparticles onto chitin nanofibers can be attributed to the strong interaction between FeS, Fe3O4 nanoparticles and chitin.

Figure 5. Photoelectron spectra of chitin and Fe/chitin (a) Fe 2p; (b) S 2p; (c) Fe 2p; (d) C1s; (e) N1s; (f) O1s.

The narrow band gap semiconductors FeS and Fe3O4 showed strong infrared absorption ability and were good photothermal conversion materials. Figure 6 showed the desalination performance of FeS/chitin and Fe3O4/chitin films with different particle proportions. As shown in Figure 6a, under nearly 10 sunlight (9.84 kW/m²), the desalination efficiency was basically positive correlation linear to the ratio of FeS and Fe3O4 and reached the maximum when the the load of FeS or Fe3O4 was in the range of 30~40%. In addition, we also tested the solar evaporation performance under the sun (1 kW/m²), as shown in Figure 6b and also randomly tested the desalinated water (Table 2), agreeing with the domestic water standard. Figure 7 showed the thermal imagings of FeS/chitin and Fe3O4/chitin composites with different proportions. Seen from the diagram, the temperature reached the maximum when the load of FeS or Fe3O4 in the range of 30~50%. Chitin film had a maximum temperature of 47.4 °C, in contrast to 63.48 °C for FeS/chitin film and
63.07 °C for Fe$_3$O$_4$/chitin film, probably because FeS and Fe$_3$O$_4$ can absorb a lot of infrared light and convert it into heat, thus improving utilization efficiency of the infrared ray. On the other hand, the structures of FeS/chitin and Fe$_3$O$_4$/chitin composites are stable after applications (Figure S1). Therefore, it is of guiding significance for other solar catalytic materials to study solar desalination.

Figure 6. The desalination efficiency of blank, chitin, FeS/chitin and Fe$_3$O$_4$/chitin films (a) and a graph of evaporation performance under the sun (b).

Table 2. Desalinated water salinity results.

| Sample         | Cl (mg/L) | NO$_2$/NO$_3$ (mg/L) | SO$_4$ (mg/L) |
|----------------|-----------|----------------------|---------------|
| Seawater       | 14495     | 819                  | 4605          |
| FeS/Chitin     | 5.460     | 4.060/1.90           | 4.153         |
| Fe$_3$O$_4$/Chitin | 7.329     | 4.097/0.18           | 10.765        |

Figure 7. Thermal imaging of chitin and FeS/chitin and Fe$_3$O$_4$/chitin with different loading ratios: (a) maximum temperature of chitin, (b) FeS/chitin and (c) Fe$_3$O$_4$/chitin. Linear diagram of (d) FeS/chitin and (e) Fe$_3$O$_4$/chitin.

Remarks: Hygienic standards for drinking water: chloride (250 mg/L), sulfate (250 mg/L), nitrate (as N, 10 mg/L). FeS/chitin and Fe$_3$O$_4$/chitin films with 40% loading were chosen for salinity test after water desalination.
4. Conclusions

FeS/chitin and FeSO₄/chitin composite films were prepared by a series of purification and dissolution processes. By changing the concentration of FeS and FeSO₄, it has been proven that a Fe base mass of 30wt% can achieve the best desalination effect. Due to the hierarchical pore structure of the film, excellent photothermal performance and good hydrophilicity, their water evaporation rates under one sun reach 1.47 kg/m²/h and 1.55 kg/m²/h, respectively. Under about 10 suns, the highest desalination efficiencies of FeS/chitin and FeSO₄/chitin are respectively 90% and 74%, and their salinity also meets the World Health Organization drinking water standards. These results indicate the potential of chitin-based nanomaterials as high-efficiency solar absorbers to produce fresh water. This work provides a green synthetic route for the construction of a mixture of natural polymers and black water-absorbing materials, and demonstrates its application potential in industrial seawater desalination.

Supplementary Materials: The following are available online at https://www.mdpi.com/.../Figure S1: SEM images of FeS/chitin and FeSO₄/chitin films after applications.

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