Solar-driven steam generation on nitrogen-doped graphene in a 2D water path isolation system

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Abstract

Solar-driven steam generation using photothermal materials is a green and promising approach to address clean water scarcity. In this work, the graphene oxide (GO) was synthesized by an improved Hummers method, and then GO was reduced by hydrothermal method and modified with nitrogen, called as NRGO. Then, we performed UV-VIS-NIR, XRD, Raman, FTIR and SEM characterization. In order to reduce the heat loss of bulk working liquid and improve the solar-to-heat conversion efficiency, the isolation system with 2D water path was used. The efficiency test of steam generation was performed under the solar intensity of 1000 Wm$^{-2}$ for 1 h, and the photothermal conversion efficiency of reduced graphene oxide (RGO) and nitrogen-doped graphene (NRGO) was 73% and 87%, respectively. It was found that NRGO owned better solar absorption efficiency, lower thermal conductivity and porosity. NRGO was combined with insulated isolated system which can reduce the heat loss of bulk water, to improve photothermal conversion efficiency.

1. Introduction

Water is the basic element of life, however, in the next few decades, the supply of clean water will be nervous, with the rapid growth of global population [1, 2]. The existing technologies can alleviate the problem of water shortage, but it will intensify the energy problem and cause detrimental effects on the environment. Therefore, many researchers have made great efforts on clean water production technologies [3–5], such as driving steam generation by solar power—using sunlight as a renewable energy source with little impact on the environment [6]. Solar-driven steam generation has emerged as a highly efficient solar-thermal process to harness abundant clean solar energy for a variety of important applications, such as clean water production. The photothermal conversion efficiency is determined by the photothermal materials that absorb solar energy. Some researchers have made great efforts to find materials with broad solar absorption spectrum and high conversion efficiency [7]. At present, photothermal materials mainly include the following categories: precious metal materials [8, 9], semiconductor materials [10–12], organic molecular materials [13] and carbon-based materials [3, 14–17]. Due to low cost, richness, excellent solar absorption capacity and high efficiency of photothermal conversion, carbon-based materials have a good development prospect and application space, such as distillation water desalination and sanitization [18].

The conversion efficiency of solar energy to steam in traditional solar driven steam generation system is low, and a large amount of funds and land resources need to be invested, which result in the universality being limited. New and efficient solar driven steam generation system can drive water evaporating without complex optical concentrator. According to the position of photothermal material placed in liquid medium, evaporation system can be classified into three types, namely volumetric system [19–21], interfacial system [8, 22, 23] and isolation system [14, 24, 25]. The volumetric system and the interfacial system respectively mean that the solar absorbing material is dispersed inside and on the surface of the bulk working liquid. In volumetric system, the bulk working liquid is inevitably heated and prone to radiation, conduction and convection heat loss, resulting in low efficiency of steam generation. The interfacial system can limit the heat energy of solar energy conversion.
to the air/liquid interface, and only the water at the interface is heated, thus reducing heat loss. In order to further improve the solar-to-heat conversion efficiency and evaporation generation, the interfacial system can be improved to separate the solar absorbing materials from the bulk working liquid to form an isolated system, to achieve the minimum effect of heat loss, such as umbrella-shaped structures and a 2D water path [25, 26].

In photothermal application, a variety of carbon-based materials which structures are from 1D to 3D have been designed and manufactured, such as 1D carbon nanotubes (CNTs) on silica [27] and a large area of 2D graphene materials [28], which have been widely used for solar steam generation, sewage treatment and seawater desalination. Graphene is a two-dimensional nanomaterial with high strength, high thermal conductivity and large surface area, and its thermal conductivity can be adjusted by chemical doping. However, due to its hydrophobicity and brittleness, graphene cannot be used for steam production alone. Therefore, steam generation of porous graphene can be completed through nitrogen doping or hydrophilic groups [29]. Different structures of graphene materials include GO films, vertically aligned and interconnected graphene [30], composite aerogels of RGO [31, 32], layered graphene foams [33] and expanded graphite foams [34], etc., show excellent evaporation efficiency due to their favorable structural characteristics. In addition, other carbon-based materials, such as hollow carbon beads [35, 36], carbon black [37–39], carbon fabric [40], carbon sponge [41], carbon foam [42–44] and coke derived carbon [45], have also been explored for steam generation. The researchers also produced biofoam/RGO [46], CNT/RGO [14, 47, 48], CNT/wood [49], GO/wood [50] and polydopamine (PDA)/wood [51] complexes, carbonized mushrooms [25] and carbonized wood [52, 53] to achieve broadband absorption, photothermal conversion and thermal localization. When the sunlight shines on the carbon-based material, the electrons are excited from valence band to conduction band. The excited electrons are relaxed by electron-phonon coupling, so the absorbed solar energy is transferred from the excited electrons to the vibration of the whole lattice, resulting in the rise of material temperature [54].

In this work, we prepared GO, RGO and NRGO and characterized them by XRD, SEM, Raman, etc. Then, the photothermal material was placed on an adiabatic body (such as polystyrene foam) with only closed pores, and the thermal insulation was wrapped with hydrophilic fiber. The water was sent to the heating surface through the capillary core absorption in the fiber.

2. Experimental

2.1. Graphene oxide preparation

Graphene oxide (GO) was prepared from 325 mesh natural flake-like graphite by an improved Hummers method [55, 56]. The synthesis steps can be divided into three stages: low temperature stage (1), medium temperature stage (2) and high temperature stage (3), as shown in figure 1.

1. 1.0 g of graphite powder was added to 23ml of concentrated sulfuric acid under stirring in an ice bath. NaN3 (0.5g) was also added to the mixture. After that, 3.0g of KMnO4 was added slowly and the solution was stirred in ice bath for at least 2 h.

2. The beaker was transferred to a 35 °C water bath heated up in advance and stirred for 2.5 h. After the reaction, 46 ml of deionized water was added to end the medium temperature reaction.

3. The beaker was transferred to a 95 °C oil bath and stirred for 15 min. After the reaction, 140 ml of warm deionized water was added, and then an addition of 10 ml H2O2 (30%) was used to make the mixture bright yellow. After the mixture was kept still, the supernatant was poured out and precipitation was washed with 1:10 dilute HCl, and then the product was centrifuged with distilled water. After ultrasonic dispersed for 40 min, the oxidized expanded graphene was freeze-dried to get GO products.

In order to obtain a GO film, 0.4 g of GO powder was dispersed into 100 ml of deionized water by ultrasonic, and then filtered by sand core funnel and dried to form a film at 50 °C.

2.2. Preparation of nitrogen-doped reduced graphene (NRGO) and reduced graphene oxide (RGO)

GO (81 mg) and urea (24.3 g) were added to 70 ml of deionized water, and then sonicated for 3 h. The solution was transferred into an autoclave and kept at 180 °C for 10 h. After the generated product was cooled, it was filtered and washed to a pH of 7. Finally, NRGO was obtained by freeze drying [57], as shown in figure 2. The preparation of RGO was similar to the above, except that urea was not utilized.

2.3. Characterization

Scanning electron microscopy (SEM) images of them were acquired by using a field emission microscope (S-4800, Hitachi). X-ray diffraction analysis (XRD) images of them were assessed by using a ray diffractometer.
(Bruker_AXS D8 Advance), and the scanning angle was 5 ~ 80°, the scanning speed was 0.25° per second, and the scanning time was 6 min. The optical properties of them were characterized by using a double-beam ultraviolet-visible-near infrared (UV-VIS-NIR) spectrophotometer (CARY5000) and the spectral range was 250 ~ 2500 nm. Raman spectroscopy test was carried out using a Raman spectrometer model (LabRAM ARAMIS) with a laser wavelength of 532.05 nm. In addition, the Fourier infrared spectrum of the samples were detected by Fourier transform infrared spectroscopy (IRA-1S WL). The surface evaporation temperature was obtained by an infrared camera (Fluke Ti50FT).

2.4. Building of the 2D water channel evaporation system
We wrapped the adiabatic foam with medical gauze to form a 2D water path, and evenly applied the same amount of NRGO and RGO powder to the top of the foam respectively. The moisture transfer of gauze and water evaporation on the material surface formed a dynamic balance, and the foam can play the role of carrier, as shown in figure 3.

2.5. Building of experimental device
The test bench mainly consisted of three parts, namely the light source system, the steam generation system and the measurement acquisition system. The light source system used artificially simulated solar emitters to provide stable light energy, as shown in figure 4(a). The experimental container used foam paper and cotton as the insulation layer on the outer wall of the beaker to reduce the heat loss of the water in the beaker, thereby improving the photothermal efficiency and steam generation efficiency. The reflective tin foil was wrapped around the outer wall of the container to prevent external light from entering the water in the beaker, thereby reducing external interference, as shown in figure 4(b).
2.6. Photothermal performance test

2.6.1. Solar absorption capacity

The sunlight trapping properties of the material include two factors, namely the absorption range of the solar spectrum and the absorption intensity of each wavelength. Therefore, in order to improve the solar-to-heat conversion efficiency, solar absorption materials are required to have high absorption rate, minimum transmittance and reflectivity in the solar spectrum.

Solar absorption rate is a measure of material’s ability to absorb solar radiation, which depends on the ratio of absorbed solar radiation to total incident radiation. Equation (1) describes the total solar energy absorption rate $\alpha(\theta)$ for the incident angle $\theta$ is [58]:

$$
\alpha(\theta) = \frac{\int_{\lambda_{\text{min}}}^{\lambda_{\text{max}}} [1 - R(\theta, \lambda)] A(\theta, \lambda) d\lambda}{\int_{\lambda_{\text{min}}}^{\lambda_{\text{max}}} A(\lambda) d\lambda}
$$

where $\lambda_{\text{min}}$ and $\lambda_{\text{max}}$ are 0.3 mm and 2.5 mm respectively, and $\theta$ is the angle of incidence of light measured from the surface normal of the absorber. $A(\lambda)$ is the solar spectral irradiance related to wavelength. $R(\theta, \lambda)$ is the total reflectance at wavelength $\lambda$.

2.6.2. Steam generation efficiency

For maximum evaporation efficiency, the heat generated by the solar absorber should be used exclusively for water evaporation. In practice, however, some heat will be lost to the vessel and surroundings by heating the bulk water or by heat conduction, convection and radiation.

The mass loss of water is measured by using solar simulators, and the energy used to evaporate water is calculated. Efficiency is calculated as equation (2) [17]:

![Figure 3. Two dimensional water channel loaded with photothermal material powder.](image)

![Figure 4. (a) Solar simulator schematic; (b) Experimental container: 1. reflective tin foil; 2. thermocouple hole; 3. cotton + foam paper; 4. pressure sensor; 5. beaker.](image)
\[ \eta = \frac{m(H_{LV} + Q)}{E_{in}} \]  

where \( m \) is the mass loss rate of water (kg m\(^{-2}\)h\(^{-1}\)) (with the dark evaporation over the samples subtracted, 0.32 kg m\(^{-2}\)h\(^{-1}\)), \( H_{LV} \) is the latent heat for evaporation of water (\( H_{LV}(T) = 1.91846 \times 10^6 [T/(T - 33.91)]^2 \) \( \text{kJ kg}^{-1} \)), \( T \) is the temperature of evaporation(K), \( Q \) is the sensible heat for increasing water temperature (\( Q = c(T - T_0) \) kg\(^{-1}\)), \( c = 4.2 \text{ J g}^{-1} \text{K}^{-1} \), \( T_0 \) is the initial temperature of water and \( E_{in} \) is the incident optical power on the solar absorber(kg m\(^{-2}\)h\(^{-1}\)).

### 3. Results and discussion

#### 3.1. Characterization

By detecting the reflection spectrum and transmission spectrum of the sample, the absorption spectra was calculated. As shown in figure 5(a), in 250 nm to 2500 nm wavelength range of GO film in the wet status, the transmittance of ultraviolet visible light was about 0%, and the reflectance was less than 10%. Thus, the absorption of ultraviolet visible wavelengths (250 nm to 790 nm) was more than 90%, and the absorption of infrared wavelengths (790 nm to 2500 nm) decreased to 80% with increasing wavelength, as shown in figure 5(b).

X-ray diffraction is an analytical method to study the crystal structure. The peak of GO was observed in the XRD pattern at 10\(^0\) and the layer spacing of GO was calculated as \( d = 0.847 \text{ nm} \), while the layer spacing of natural graphite powder was only 0.335 nm. The crystal shape of the oxidized graphite on the surface was destroyed, and the new crystal shape was produced, which expanded the spacing of the oxidized graphite layer to more than twice. Mainly because of the existence of hydroxyl group (—OH), carboxyl group (—COOH) and other groups in the oxidation process, the interlayer spacing became larger, that facilitated the entry of other molecules to form graphene composites, such as nitrogen-doped graphene. After 1 h of illumination, the diffraction peak intensity of about 10\(^0\) significantly weakened, and a weak wide valley peak appeared at about 25\(^\circ\), as shown in figure 5(c). The oxidation degree of GO on the surface was weakened, and most of it had been reduced to graphitization.

The diffraction peaks of NRGO and RGO appeared in around 25\(^\circ\) [C(002)] and 10\(^\circ\) peak disappeared, indicating that hydrothermal reaction reduced GO by partially removing oxygen-containing groups on the surface and restacking them into disordered graphitized materials, as shown in figure 5(d). The C(002) peak of NRGO was weaker but larger than that of RGO, which can be attributed to the less re-graphitization of NRGO, because nitrogen atoms were bound to the carbon skeleton of graphene. Moreover, the angle corresponding to the diffraction peak of NRGO was larger than that of RGO, indicating that urea can more completely make GO reduced in NRGO, and N dopant can partially inhibit the re-graphitization of graphene sheets in RGO.

It can be seen from the FTIR diagram that, compared with GO, the intensity of hydroxyl peak (O–H) in 0.5 h and 1 h of illumination decreased with the increase of illumination time, and the oxygen-containing groups were gradually not obvious, as shown in figure 5(e). After reduction by hydrothermal reaction without urea, the FTIR spectrum of RGO showed a weak C–O peak and a weakened C–OH tensile. These results can be inferred that after hydrothermal treatment, a small portion of C–O and O–H groups distributing on the carbon surface disappear, and the reduction degree of GO was limited [59]. FTIR spectrum of NRGO showed sp\(^2\) C–N bond between 1590 ~ 1620 cm\(^{-1}\) and C–N bond at 1456 cm\(^{-1}\) [60], as shown in figure 5(f). It was shown that during the process of hydrothermal treatment, the oxygen substance in GO was reduced and N was doped into the graphene framework.

Raman spectroscopy showed that all samples had two distinct characteristic peaks, which were located at D peak (~1325 cm\(^{-1}\)) and G peak (~1580 cm\(^{-1}\)). Generally speaking, D peak was related to lattice defect and used to represent the disorder degree of graphite carbon, while G peak was derived from the planar stretching vibration of carbon atom sp\(^2\) hybrid. The intensity ratio of D peak to G peak (I\(_D\)/I\(_G\)) was usually used to characterize the defect density of graphene. The smaller the ratio was, the lower the disorder degree was. For highly oriented graphite, the ratio was close to 0 [61]. As shown in figure 6, the I\(_D\)/I\(_G\) value of RGO and NRGO samples were 1.21 and 1.27 respectively, both of which were higher than the I\(_D\)/I\(_G\) value of GO (1.03), indicating that the average size of graphene sheets became smaller, because GO was reduced and re-graphitized, leading to more defects in RGO and NRGO. However, the introduction of nitrogen can increase the defect site of the material, so that the I\(_D\)/I\(_G\) value of NRGO was higher than that of RGO. Raman diagram showed that RGO and NRGO had low degree of graphitization and large disorder, mainly amorphous carbon [52]. As amorphous carbon did not have a long-term orderly structure, its thermal conductivity was low, thus reducing the heat loss in the process of photothermal conversion was conducive to the concentration of heat energy in the solar absorption material for local heating of water.
Scanning electron microscopy (SEM) images showed that the pores in the nitrogen-doped graphene were randomly distributed, interconnecting into a three-dimensional porous network, as shown in Figure 7. Since the micron size of the aperture was more conducive to the core absorption effect of water than nanoscale hole, this structure was conducive to the generation of steam.

### 3.2. Solar-driven steam generation experiment

#### 3.2.1. Mass loss test

We used the designed insulation structure to conduct evaporation experiments at 1000 Wm$^{-2}$. After 1 h illumination, the mass loss of pure water was 1.53 g. Compared with the mass loss of photothermal materials loaded on the water surface, it indicated that water was not strong in solar energy absorption. In order to improve the efficiency of steam generation, photothermal materials needed to be used for solar absorption, heat conversion and heat storage. In addition, we used foam as a unique thermal barrier, and the surface was covered with water absorbent fiber material. Moreover, we compared the amount of steam generated by the GO film and its foam wrapped in the gauze, as shown in Figure 8(a). Comparing results verified that the two-dimensional water channel method can increase steam generation, because the contact area between water body and the solar absorption material in the isolation system was smaller than that of the interfacial system. After that, comparing

![Figure 5](image-url)

*Figure 5.* (a) Reflection and transmission spectrum of GO; (b) Absorption spectrum of GO; (c) XRD pattern of GO after 1 h illumination; (d) XRD patterns of GO, RGO and NRGO; (e) FTIR images of GO and GO illumination for 0.5 h and 1 h; (f) FTIR images of GO, RGO and NRGO.
the two-dimensional water channels of RGO and NRGO powders at 1000 W m$^{-2}$ light intensity for 1 h, the average evaporation of them was 2.43 g and 2.8 g respectively, as shown in figure 8(b). After subtracting their dark evaporation, their evaporation rate was 1.05 kg m$^{-2}$ h$^{-1}$ and 1.26 kg m$^{-2}$ h$^{-1}$. The porous materials RGO and NRGO can greatly improve the steam generation efficiency, because RGO and NRGO had certain
hydrophilicity and micron-sized pores inside, that was beneficial to generate wicking and increase the amount of steam.

3.2.2. Temperature distribution change

By detecting the temperature at the 30, 60, 90 ml positions of a 100 ml beaker, we can know the temperature distribution of water after the irradiation of 1000 Wm\(^{-2}\) for 1 h. As shown in figure 8(c), the temperature distribution of the no-load situation showed that the temperature of the lower water level increased greatly, and the heat loss transferred to the water was large. By comparing with a 2D water channel of RGO, we can know that the heat was mainly gathered in the solar absorbing material. Therefore, the heat to raise temperature of water was relatively small, even the lower water level within 1 h only increased by 3.1 °C. It verified that most of the heat generated by RGO absorbing solar energy was used to heat and evaporate water on the surface, as shown in figures 8(c) and (d).

Insulation systems using RGO powder and NRGO powder as solar energy absorbing materials were irradiated with 1000 Wm\(^{-2}\) light for 1 h, and their temperature change curves were measured and recorded as shown in figure 9. The temperature increments of NRGO at the measuring points 1, 2 and 3 were smaller than that of RGO respectively, but the insulation system of NRGO produced more steam than RGO within 1 h. Therefore, it can be seen that NRGO was more conducive to the concentration of heat for steam generation than RGO, by reducing the heat loss of heated water. At the same time, nitrogen doping can improve the photothermal conversion efficiency and increase the steam evaporation yield.

3.2.3. Efficiency calculation

A solar simulator with a constant energy output of 1kWm\(^{-2}\) was used as the light source. An infrared (IR) camera was employed to measure the temperature of the steam on the surface of the material. As shown in figure 10, the average temperature of the top surface was available. The final steam temperatures of no-load and two-dimensional water channels loaded with RGO and NRGO were 84.1 °F (28.9 °C), 89.3 °F (31.8 °C) and 91.4 °F (33.0 °C), respectively. The water evaporation rate \(m\), the latent heat \(H_f\), the temperature of evaporation \(T\) and the heat for increasing water temperature \(Q\) were known, and then calculated by equation (2), the photothermal conversion efficiency of no-load and RGO was 38% and 73%. The photothermal conversion efficiency
efficiency of the 2D water channel of NRGO was 87%, 7% higher than porous N-doped graphene sheet by CVD growth at 950 °C (80%) [29].

4. Conclusion

As a solar absorption material, GO not only had hydrophilicity, low interlayer thermal conductivity, porosity and excellent optical absorption properties, but also had the advantages of easy scale preparation, easy folding, convenient carrying and light weight, effectively reducing optical loss and improving efficiency. RGO and NRGO hydrogel or powder obtained through reduction and nitrogen-doped reduction of GO can improve the steam generation efficiency. The photothermal conversion efficiency of RGO was 73%, and that of NRGO was 87%. The experimental results showed that nitrogen doping modification of GO had better efficiency and heat conversion efficiency of the steam, because NRGO had desultorily reticular pores inside, which made the thermal conductivity lower and it was more convenient to store heat energy. In addition, its micro-pore facilitated the wicking of water and greatly increased steam generation efficiency.

Desalination is a dream that mankind has pursued for hundreds of years, but seawater desalination has not been widely used due to technical and cost constraints. In this work, the characterizations and experimental results of NRGO show that there is a large space for photothermal applications, providing a possibility for the development of portable low-cost solar desalination technologies.

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