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Preparation of a graphene oxide membrane for air purification

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Abstract

Environmental issues such as particulate matter (PM) have become more and more serious because of their detrimental effects on human health. The development of an effective way to remove PM is in high demand. In this study, a graphene oxide (GO) membrane with a large specific surface area and a continuous pore structure was prepared via a coating method. The morphology, composition and structure of the GO membrane were characterized by scanning electron microscopy (SEM), an energy dispersive spectrometer (EDS), X-ray diffraction (XRD), FT-IR spectra, X-ray photoelectron spectra (XPS) and Raman spectroscopy. The removal efficiency for PM and the influence of pollutant concentration and wind velocity on the GO membrane were studied. As a result, when the wind velocity was 0.1 m s\(^{-1}\), the PM\(_{2.5}\) removal efficiency of the GO membrane exhibited the highest efficiency (99.46%) with a low pressure drop (7 Pa) and a high quality factor (QF) of 0.75 Pa\(^{-1}\). The GO membrane maintained a high adsorption performance for a long time. The GO membrane removal efficiency was more significant when the import concentration was higher and the wind velocity was lower. In particular, SEM and EDS clearly revealed that the PM\(_{2.5}\) was successfully captured by the GO membrane. The preparation of the GO membrane involves a simple and pollution-free process, which provides a new way to deal with air pollution.

1. Introduction

Environmental problems have become increasingly serious due to the industrial revolution, rapid developments in industrialization, population expansion and urbanization [1]. As a major pollutant, particulate matter with a diameter of 2.5 \(\mu\)m or less, called PM\(_{2.5}\), can be inhaled into the lungs, and prolonged exposure can cause serious health consequences for humans [2–5]. To solve this problem, graphene has attracted enormous attention because of its excellent properties such as a large specific surface area, remarkable electrical, thermal conductivity, and high light transmittance [6–9]. It is widely used in solar cells [10], supercapacitors [11], air purification [12], gas sensors [13] and other fields.

To explore the unique properties in air purification, researchers have concentrated on more efficient technology to take advantage of graphene. Hong et al reported a new strategy to fabricate graphene-supported carbon-coated cobalt and carbon nanoshells (Co/C-GNS and CNS-GNS) via a hydrothermal method and a catalytic carbonization process. The authors studied this process for adsorbing toxic gases, and the results showed that they exhibited an extraordinary adsorption performance for toxic CO and heavy smoke [14]. Liang et al synthesized an amine-functionalized graphene aerogel through gelation and in situ reduction of GO by using polyethyleneimine. Then, the graphene aerogel was obtained by freeze drying. The composites were used in the adsorption of HCHO, with a maximum HCHO adsorption capacity of 2.43 mg g\(^{-1}\) at the saturation state [15]. Li et al have shown that graphene oxide and polyacrylonitrile are dispersed and dissolved in N,N-dimethylformamide (DMF) used as a solvent and that all membranes can be deposited onto non-woven substrates by an electrospinning process. The authors showed that the composite membrane had a high...
filtration effect for PM$_{2.5}$ and exhibited a high efficiency of 99.97% with a low pressure drop [16]. Li et al reported that nest-like multilevel structured graphene oxide-on-polyacrylonitrile (PAN/GO) membranes fabricated by combining multi-jet electrospinning and physical bonding can be used for the filtration and separation of ultrafine particles [17]. Jeong et al demonstrated that a Transparent, Reusable and Active PM (TRAP) filter with an Ag nanowire percolation network prepared via a simple vacuum filtration method showed a very high efficiency [18].

At present, most research on graphene in the field of air purification focuses on composite materials such as mixing graphene as an additive with some organic solvents or loading it onto other substances. In this study, graphene oxide was prepared by the modified Hummers method, and the graphene oxide slurry was deposited onto non-woven substrates by coating technology to prepare a graphene oxide membrane. The effect of the non-woven fabric as a substrate on PM$_{2.5}$ is extremely small, so one can investigate the effect of pure GO on PM$_{2.5}$. In addition, in this paper, the method of coating GO onto non-woven fabric was adopted and the adsorption performance of PM$_{2.5}$ was discussed. The whole device designed by the laboratory was completely controlled by the computer, and the pollutant concentration, pressure drop and wind velocity of the GO membrane can be simultaneously measured to evaluate the adsorption performance of the GO membrane. The preparation of the GO membrane is simple and controllable, which provides a feasible way to develop air membrane purification technology.

2. Experimental section

2.1. Materials
GO was prepared by a modified Hummers method [19–21] with deionized water prepared in the laboratory. The non-woven substrates (Polypropylene, PP) with a thickness of 200 ± 1 μm and weight of 20 g m$^{-2}$ were purchased from the Xinshengli Environmental Protection Technology Co., Ltd.

2.2. Methods
2.2.1. Preparation of GO membrane
The GO solid was dispersed in deionized water and stirred for 150 min to swell sufficiently in water. Subsequently, the suspension of GO was sonicated for 90 min to prepare a 40 mg ml$^{-1}$ GO slurry by using sonicator (2000 Hz, 1500 W) and thermostat (Ultrasonic Material Disperser LC-1500 and Low Temperature Thermostat LCDC-1006F, Ningbo Licheng Co., Ltd). The setting temperature was 20 °C. Then, GO slurry was deposited onto non-woven substrates by using a typical blade coating process (Heating Type Membrane Coating Machine PF400-H, Jiangsu LEBO Science Co., Ltd). Among them, the dust-removed and dried non-woven fabric was fixed on a high-precision glass, and the glass was adsorbed on the coating machine by vacuuming. The prepared 40 mg ml$^{-1}$ GO slurry was scraped onto non-woven fabric and the membrane was formed by blade coating once [22]. In particular, the coating speed and coating membrane size were 10 mm s$^{-1}$ and 10 × 10 cm$^2$, respectively. Finally, the prepared GO membrane was naturally dried for 24 h and used for testing. The whole process was carried out at normal pressure and temperature, and the humidity range was 40%-60%. The quality of the dried GO membrane was 6.05 g m$^{-2}$, and the thickness was 200 ± 1 μm. The preparation process is shown in figure 1.

2.2.2. Characterization
The surface morphologies of the non-woven substrate, GO powder and GO membrane were characterized by field-emission scanning electron microscopy (SEM, Quanta 650 FEG). Before the SEM observation, the membrane was sputter coated with an ultra-thin layer of gold. Raman spectra were recorded using a K-Sens-532 to characterize the GO membrane, with an excitation laser wavelength of 532 nm. The structure of GO powder was characterized by XRD using a Shimadzu XRD-7000 Diffractometer ($\lambda = 1.5416$ Å). FT-IR spectra were collected using a Thermo Nicolet 6700 FT-IR spectrometer and XPS data were recorded on a PHIQUANTERA II SXM X-ray photoelectron spectrometer.

2.2.3. Adsorption of PM$_{2.5}$
The tests for the adsorption of PM$_{2.5}$ were performed in a system (figure 2) by using the technique of sandalwood-burning to simulate PM$_{2.5}$ in the air [23]. Among them, the whole device was controlled by computer. The pollution source was composed of pollutants and air. The polluted gas was transmitted to the GO membrane through the peristaltic pump and the flow meter, and the polluted gas passed through the GO membrane by means of suction. Particularly, the wind velocity used in the experimental test was 0.1 m s$^{-1}$. The GO membrane was removed for performance analysis after testing. The removal rate of PM$_{2.5}$ by the GO membrane was calculated using (C$_{0}$–C$_{1}$)/C$_{0}$ × 100%, where C$_{0}$ is the concentration (import concentration).
before the pollutant passes through the GO membrane, and $C_1$ is the concentration (export concentration) after
the pollutant passes through the GO membrane. Particularly, the entire experimental device was maintained at a
normal temperature and pressure for experimentation. The airflow used by the device did not set any pressure
for natural circulation.

3. Results and discussion

3.1. Structural characterization of GO membranes

3.1.1. SEM analysis of the GO membrane

As shown in figure 3(a), the non-woven substrates show a typical interlaced structure that forms a large number
of holes due to its disordered spatial arrangement, which provides a basis for the attachment of GO. Figure 3(b)
shows the typical structure of GO, including a large number of wrinkling and crumpling structures and a few
layers. Figure 3(c) illustrates that GO is successfully deposited onto non-woven substrates. Meanwhile, the
interspaces in the non-woven substrate fibres are filled by GO to form a complete membrane structure.
Figure 3(d) exhibits a tortuous and uneven fibre structure, which is significantly different from the neat and
smooth fibre structure shown in figure 3(a). It is fully demonstrated that not only is GO attached to the surface of
the non-woven substrates, but it also effectively wraps the fibres. Moreover, it improves adhesion and makes the
membrane structure stronger, which facilitates the deposition of air pollutants in space. Figures 3(e) and (f)
show the cross-section view of the GO membrane and non-woven substrate. From the SEM images, it can be
clearly seen that non-woven fabric is a three-dimensional loose structure. In particular, the non-woven fibres change the morphology and become more consistent because of the addition of GO. The depth of penetration of graphene into the non-woven fabric is shown in figure 3(e). However, this depth does not represent the thickness of the graphene membrane, but a large amount of membrane is formed in the space with the fibers of the three-dimensional structure. In general, GO only occupies a very thin membrane on the non-woven substrates. As a result, the whole GO membrane is a perfect combination of non-woven substrate and GO, which not only retains the characteristics of GO but also compensates for the disadvantages of a large fibre gap and poor integrity.

3.1.2. SEM and EDS analysis of PM$_{2.5}$ adsorbed on the GO membrane

Typical SEM images of the GO membrane before and after the examination of PM are shown in figures 4(a) and (b), respectively, which clearly show that a large number of white spherical substances appear in the GO membrane after the examination. Furthermore, the size of the particulate matter is different. It is found that
particulate matter less than 2.5 μm or even smaller (<0.5 μm) could also be captured according to the scale, which distributes on the surface of the GO membrane and between GO layers. In particular, it is indicated that PM2.5 is successfully captured by the GO membrane. Figure 4(c) shows enlarged detail for PM2.5, which is firmly embedded in the GO layer, with some particles located deep inside the layer. Moreover, the wrinkled and overlapping structure of GO can be clearly seen. Therefore, the SEM images fully demonstrate the adsorption characteristics of the GO membrane.

To further prove that the GO membrane successfully captured PM2.5, EDS analysis of the GO membrane before and after the experiment were carried out. The data are summarized for points 1 and 2, which are from the area shown in figure 4(d), as shown in table 1. It can be seen from point 2 that the GO membrane contains only C and O elements before the experiment. In addition to C and O elements, N and Si elements also appear at the data display point 1. Furthermore, the membrane contains a larger number of N elements due to the adsorption of a large amount of nitrogen oxides on the surface of the GO membrane after the experiment. Since PM2.5 contains elements such as N and Si, the adsorption characteristic for PM2.5 on the GO membrane is confirmed. In addition, the mass ratio of C to O at point 2 is approximately 2.52:1, which indicates that the oxidation degree is higher and contains a large number of oxygen-containing functional groups with high activity.

**Table 1.** EDS of a GO membrane before and after experiments.

| Spot                  | Element | Weight (%) | Atom (%) |
|-----------------------|---------|------------|----------|
| 1 (after experiments) | C K     | 65.7       | 70.17    |
|                       | O K     | 13.18      | 10.57    |
|                       | N K     | 20.95      | 19.19    |
|                       | Si K    | 0.18       | 0.08     |
| 2 (before experiments)| C K     | 71.63      | 77.08    |
|                       | O K     | 28.37      | 22.92    |

**Figure 4.** (a), (b) SEM images of GO membranes before and after examination; (c) SEM images showing the detail of PM2.5; (d) EDS of the selected area.
3.1.3. Raman spectral, XRD, FT-IR and XPS analysis of the GO

Raman spectra can provide further evidence for the existence of GO, so GO was characterized by Raman spectroscopy. The Raman spectra measured for the GO membrane before and after testing are shown in figure 5(a). There are two distinct peaks, corresponding to the D peak at 1347 cm\(^{-1}\) and the G peak at 1587 cm\(^{-1}\). The \(I_D/I_G\) ratios before and after the test are 0.999 and 0.998, respectively. Among these peaks, the D peak is caused by sp\(^3\) carbon atoms with a disordered carbon structure or defective structure, and the G peak is caused by the vibration of sp\(^2\) hybrid carbon atoms from the aromatic structure\(^{[24]}\). Generally, the defect degree of nanomaterials is expressed by \(I_D/I_G\). Moreover, the higher the ratio, the greater is the GO activity. By comparing the peak positions, no blue shift is observed between the two peaks before and after the experiment. Moreover, the change in the \(I_D/I_G\) value is very small, which indicates that the GO membrane has good stability and is less affected by the environment. Above all, the stable structure is accompanied by excellent adsorption performance, which also provides a basis for the long-term work of the GO membrane.

Figure 5(b) is the XRD of GO, which yields a strong diffraction peak at \(2\theta = 10.8^\circ\), corresponding to the crystal plane of GO (001) and the layer-to-layer distance between GO sheets (0.819 nm), indicating that the graphite is oxidized to introduce an oxygen-containing functional group. GO yields an infrared spectrum (figure 5(c)) containing peaks at 1061, 1224, 1607, corresponding to C–O, C–O–C, and C=C bonds, respectively. In addition, the peaks at 1723, 3190 are caused by the stretching vibration of the carboxyl group COOH and the hydroxyl group O–H, respectively. To further demonstrate the degree of GO oxidation, the C 1s XPS results of GO is shown in figure 5(d). The spectrum of GO can be deconvoluted into four components, of which the main peaks at 284.9, 285.7 and 286.8 eV correspond to C–O–C, C=O, O–C=O species, respectively, and the peak at 283.5 eV caused by C–C bond. The results of XRD, FT-IR and XPS show that there are a large number of oxygen-containing functional groups in GO materials prepared in the laboratory, which is important for PM\(_{2.5}\) removal.

3.2. Adsorption performance of the GO membrane

A dried GO membrane was used to measure its adsorption performance and calculate the removal rate. The pollutants and air cylinders were turned on, and the concentration of PM\(_{2.5}\) was controlled to maintain a
relatively stable concentration range during the experiment, which was recorded every 10 min and continuously measured for 60 min. The removal rates for the non-woven substrates and GO membrane are shown in figures 6(a) and (b), respectively.

As shown in figure 6(a), the removal rate for pure non-woven substrates is only 8.72%. However, even if the import concentration fluctuates, the export concentration of the polluted gas passes through the GO membrane almost unchanged from figure 6(b). Specifically, the removal rate of PM2.5 reaches 99.46%, increasing by 90% compared to the pure non-woven substrates.

### 3.2.1. Effect of the import concentration of pollutants on the adsorption properties of the GO membrane

The import concentration was set to different dimensional values (500–700 μg m$^{-3}$, 700–900 μg m$^{-3}$, over 900 μg m$^{-3}$) by changing the flowing rate, which was used to study the removal rate of PM$_{2.5}$ by the GO membrane and its mechanism. From figure 7(a), it can be seen that the removal rate increases with increasing import concentration. Furthermore, the highest removal rate is 97.17% when the import concentration is above 900 μg m$^{-3}$, and the removal rate is 95.09% when the concentration is low. To be sure, with increasing import concentration, more PM$_{2.5}$ is adsorbed on the GO membrane. However, the export concentration is almost unchanged, which will be affected by the natural environment so that the removal rate will increase.

In general, serious pollution occurs when the PM$_{2.5}$ concentration is more than 300 μg m$^{-3}$, which is the most harmful to humans [25]. Therefore, a concentration of PM$_{2.5}$ ($>300$ μg m$^{-3}$) was chosen in the experiment, and the data were recorded every 0.5 h for 24 h. Additionally, the long-term working efficiency of the GO membrane was investigated. Figure 7(b) shows the long-term performance of the GO membrane. The GO membrane still maintains a high PM$_{2.5}$ removal efficiency of 97%–99% after 24 h with low pressure drop of 7 Pa–9 Pa. As a result, with high removal efficiency and excellent long-term performance, the remarkable stability of the GO membrane is further confirmed; this is in agreement with the previous results obtained from the Raman spectra.

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**Figure 6.** Removal rate of PM$_{2.5}$ for (a) non-woven substrates and (b) GO membrane.

**Figure 7.** (a) Effect of the import concentration of pollutants on the removal rate; (b) Long-term working removal rate of GO membrane.
3.2.2. Effect of wind velocity on the adsorption properties of the GO membrane

How wind velocity influences the pressure drop and removal rate was systematically investigated by adjusting the transformer and changing the wind velocity. The wind velocity (0.1 m s\(^{-1}\), 0.2 m s\(^{-1}\) and 0.3 m s\(^{-1}\)) and the pollutant import concentration (550 μg m\(^{-3}\), 500 μg m\(^{-3}\) and 440 μg m\(^{-3}\)) were measured by ignoring slight fluctuations. The pressure drop and the removal rate with different wind velocities are shown in figures 8(a) and (b), respectively.

In addition to the removal rate, another important ideal parameter is low pressure drop, which reduces energy consumption\(^{[26]}\). Figure 8(a) shows that with increasing wind velocity, the pressure drop increases gradually. For example, when the wind velocity is 0.3 m s\(^{-1}\), the pressure drop is 20 Pa, and that with 0.1 m s\(^{-1}\) is only 7 Pa. In total, the GO membrane exhibits an excellent pressure drop and a higher quality factor (QF). The values of QF can be defined by the following formula \(-\ln(1-E)/\Delta p\), where \(E\) is the removal rate and \(\Delta p\) is the pressure drop\(^{[27–29]}\). The QF of the GO membrane reaches 0.75 Pa\(^{-1}\) when the PM\(_{2.5}\) removal rate is 99.46% (maximum removal rate of GO membrane) and the related pressure drop is 7 Pa at the wind velocity of 0.1 m s\(^{-1}\).

As shown in figure 8(b), with increasing wind velocity, the removal rate of PM\(_{2.5}\) by the GO membrane decreased. Among these data, when the conditions are \(V = 0.1 \text{ m s}^{-1}\) and \(C_0 = 550 \text{ μg m}^{-3}\), the removal rate is 96.36% and the QF is 0.47 Pa\(^{-1}\). The main reason for this is that the interaction time between PM\(_{2.5}\) and the GO membrane is not enough because of the large wind velocity, and PM\(_{2.5}\) cannot be captured efficiently. Therefore, increasing the residence time of PM\(_{2.5}\) in the GO membrane has a higher removal rate at a low flow rate.

**Figure 8.** (a) Relationship between the pressure drop and wind velocity; (b) Effect of the wind velocity on the removal rate.

**Figure 9.** Removal mechanism of PM\(_{2.5}\) by a GO membrane.
3.3. Mechanism analysis
The GO with few layers prepared by the modified Hummers method had a large specific surface and rich oxygen-containing functional groups. Additionally, the GO membrane with micron-scale stacks was obtained by coating technology. The removal mechanism of PM$_{2.5}$ by a GO membrane is shown in figure 9.

First, by investigating the concentration and wind velocity of pollutants, the removal rate of PM$_{2.5}$ by the GO membrane was found to mainly depend on adsorption because of the existence of Van der Waals force. A high import concentration and low wind velocity provide sufficient contact time and sufficient contact area for the GO membrane. In addition, the fiber wrapped by GO increases the specific surface area to facilitate adsorption of PM$_{2.5}$. Second, micron-scale sheets are stacked tightly, increasing the transmission path and providing a shortcut for the entry of PM$_{2.5}$. There are a large number of sub-micron porous structures that contribute to the smooth passage of airflow. Therefore, the GO membrane plays a certain role in pore size selection. Finally, oxygen-containing functional groups such as hydroxyl (-OH) on GO surface and functional groups such as (Si-OH)$_n$(C=O) contained in PM$_{2.5}$ will have a dipole-dipole interaction, thus forming a new bond, which can firmly adsorb PM$_{2.5}$ on GO surface. In particular, PM$_{2.5}$ and GO have a π-π stacking action and are thus adsorbed on the GO surface [30, 31]. In summary, the removal mechanism for the GO membrane is not based on reducing porosity and increasing compactness but on the formation of a large number of transport channels through the stacking of GO sheets at the micron level, increasing pore size selection, and the interaction of GO itself with a large specific surface area and rich oxygen-containing functional groups, i.e., adsorption, filtration and capture of chemical adsorption sites.

4. Conclusions
Graphene oxide was prepared by the modified Hummers method in the laboratory, and GO membrane was prepared by coating technology for air purification. The adsorption properties of the GO membrane were investigated.

1. SEM images show that the GO membrane plays the role of adsorbent and filter and can successfully capture PM$_{2.5}$. After 24 h of continuous measurement, the removal rate can still be maintained at 97%–99%.

2. The import concentration, wind velocity and pressure drop of pollutants were studied as influence factors. The outcome shows that the GO membrane removal efficiency is more significant when the import concentration is higher and the wind velocity is lower, with a lower pressure drop observed while maintaining high removal efficiency. Through fully simulating air pollution and testing, the GO membrane shows a high PM$_{2.5}$ removal rate of up to 99.46%, low pressure drop of 7 Pa, and a high QF of 0.75 Pa$^{-1}$ under a wind velocity of 0.1 m s$^{-1}$.

3. The GO membrane is highly efficient and its preparation is green and pollution-free, which provides technical support for research and application in the field of air purification.

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