Multilayer-structured fibrous membrane with directional moisture transportability and thermal radiation for high-performance air filtration

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Abstract: The demand of high-performance filter media for the face masks is urgent nowadays due to the severe air pollution. Herein, a highly breathable and thermal comfort membrane that combines the asymmetrically superwettable skin layer with the nanofibrous membrane has been fabricated via successive electrospinning and electrospraying technologies. Thanks to high porosity, interconnected pore structure, and across-thickness wettability gradient, the composite membrane with a low basis weight of 3.0 g m$^{-2}$ exhibits a good air permeability of 278 mm s$^{-1}$, a comparable water vapor permeability difference of 3.61 kg m$^{-2}$ d$^{-1}$, a high filtration efficiency of 99.3%, a low pressure drop of 64 Pa, and a favorable quality factor of 0.1089 Pa$^{-1}$, which are better than those of the commercial polypropylene. Moreover, the multilayer-structured membrane displays a modest infrared transmittance of 92.1% that can keep the human face cool and comfort. This composite fibrous medium is expected to protect humans from PM$_{2.5}$ and keep them comfortable even in a hygrothermal environment.

Keywords: electrospinning, air filtration, wearing comfortability, thermal radiation, nanofiber membrane

1 Introduction

Severe air pollution is currently one of the global issues that threaten human beings, which is mainly due to the emissions from on-road vehicles, industrial production activities, and dust (1,2). Therefore, cost-effective strategies to remove PM$_{2.5}$ (particle diameter <2.5 μm) from air are of special importance (3,4). Wearing a face mask is considered as one of the most direct and effective methods to protect the human body. The core filter media of the face masks are usually composed of various fibers due to their distinctive structures and good processability. However, the melt-blown fiber-based filter media cannot satisfy the requirements of a face mask, owing to their limited capture property toward ultrafine particle and slow moisture evaporation rate (5), thus causing human body to feel uncomfortable and even dangerous during respiration. Nanofibers supply a dramatic promotion in PM$_{2.5}$ capture efficiency and provide a sharply decreased basis weight (6). Additionally, nanofiber-based filter medium could be easily functionalized to obtain special properties, such as antibacterial activities (7), dye scavenging (8), and easy cleaning performance (9).

Benefiting from the tunable nanofiber structure, interconnected open pore structure, and high porosity, the electrospun nanofibrous membrane (NFM) was considered as a suitable filter medium for cost-effectively capturing PM$_{2.5}$ (10–12). Based on these, a variety of electrospun fibrous media for air filtration have purposely been developed, including polyacrylonitrile (PAN) and polylactic acid (13,14). In addition, inorganic SiO$_2$ nanoparticles (SiO$_2$ NPs) possess the properties of good charge storage ability and stable electric field as reported in a previous study (10). Electrospinning could integrate the electret process and fiber construction into one process. Thus, by combining the electrospinning of right type of polymer and SiO$_2$ NPs, a desired composite membrane with a hierarchical structure and good performance could be fabricated. However, nearly no effort has been put to the study on the wearing comfortability of an electrospun composite membrane, especially on the relationship between the membrane structure and wearing comfortability. Therefore, the challenge remains in developing NFMs with high filtration performance and creditable wearing comfortability. Inspired by the features of moisture-wicking technologies and push–pull effect, NFMs with an across-thickness wettability gradient have been fabricated to study the effects of surface structure and wettability on the directional moisture transport process. Based on these, a composite NFM which possesses...
asymmetric wettability may be a promising candidate for meeting the demands of high-performance face masks.

In this study, a sequential electrospraying approach was demonstrated for the construction of an asymmetrically superwettable skin layer that was integrated with the electrospun PAN/polyetherimide (PEI) NFM substrate to obtain a multilayer-structured nanofiber membrane (SNFM) with high PM$_{2.5}$ capture performance, desirable air permeability, and good radiative cooling properties. The morphology, pore structure, breathability, and water vapor transmission rate (WVTR) can be readily controlled by simply tailoring the polymer concentration and fiber basis weight. Notably, bi-functions of low air permeability resistance and smooth directional moisture transfer were observed for the composite membrane with an across-thickness wettability gradient. Moreover, the SNFM with an outstanding radiative cooling performance is expected to significantly improve the thermal radiative dissipation in extreme environmental conditions.

2 Materials and methods

2.1 Materials

PAN powders (P823209, $M_w = 85,000$) were obtained from Shanghai Macklin Biochemical Technology Co., Ltd, China. PEI (Ultem 1000, $M_w = 10,00,000$) was provided by General Electric Company, USA. Trimethoxy(heptadecafluorotetradecyl)triethoxysilane (FAS, C$_{38}$H$_{73}$F$_{17}$O$_3$Si, 97%) was bought from Sigma-Aldrich. $N, N$-Dimethylformamide (DMF, AR 99.7%) was supplied by Shanghai Chemical Reagents Co., Ltd, China. Nanofumed silica (SiO$_2$, hydrophilic-200, 7–40 nm) was purchased from Shanghai Aladdin Chemical Co., China. All chemical reagents were used as received without any further processing.

2.2 Preparation of solutions

DMF was used as the solvent to prepare the PAN (10 wt%) and PEI solutions (12 wt%), which were constantly stirred for 10 h with a stirring rate of 800 ± 50 rpm at room temperature, separately. A PEI solution (6 wt%) containing 6 wt% superhydrophobic silica nanoparticles (SiO$_2$ NPs) was obtained through the following procedure: first, 0.036 g of SiO$_2$ NPs was uniformly dispersed in 9.4 g of DMF under continuous stirring with a rate of 800 ± 50 rpm at room temperature for 30 min, followed by sonication treatment for another 30 min, and then 0.072 g of FAS was added to the above mixture and stirred at 60°C for 6 h. The detailed preparation process of FAS-modified superhydrophobic SiO$_2$ NPs (F-SiO$_2$) is shown in Figure 1a. Finally, 0.6 g of PEI was injected into

![Figure 1](image-url)
the prepared suspension followed by stirring for 8 h. A PAN solution (5 wt%) containing SiO$_2$ NPs (4 wt%) was also obtained via the same method as that of the PEI solution containing F-SiO$_2$ NPs.

2.3 Fabrication of multilayered fibrous membranes

First, PAN and PEI fibers were separately fabricated via electrospinning with a feed rate of 2 mL h$^{-1}$, and the obtained double-layered composite membranes were denoted as PAN/PEI NFMs. Additionally, the skin layer was prepared by electrospaying the resultant SiO$_2$@PAN and F-SiO$_2$@PEI solutions with the same feed rate of 0.3 mL h$^{-1}$ and the generated fibers were continuously deposited on the electrospun PAN and PEI membranes. The parameters used during the fabrication process were as follows: the applied voltage was fixed at 25 kV, the tip-to-collector distance was fixed at 15 cm, the temperature was 25 ± 2°C, and the relative humidity was 45 ± 5%.

2.4 Characterization

The morphology and structure of the as-prepared fibers were characterized by a scanning electron microscope (SEM; Phenom ProX, Phenom World, USA). A bubble point method was applied to test the pore size distribution (PSD) of the membranes on a Pore Size Meter (PSM165; Topas GmbH, Dresden, Germany). A standard solution with a surface tension of 16 dynes/cm was used as the wetting liquid. The prepared membrane was immersed in this wetting liquid, and then the pore size was tested with the pressure ranging from 0 to 58 psi. The filtration performance was measured using an automated filter tester (Filter Media Test RIG AFC 131; Topas GmbH, Dresden, Germany). The tester could deliver charge-neutralized NaCl aerosol particles with a diameter of 300–500 nm. At a flow rate of 33 L/min, the NaCl aerosol particles entered pipe of the filter tester and passed through the filter medium with a diameter of 18 cm which was sandwiched between the upper and lower pipes of the filter tester before the test beginning. The air flow resistance was tested with two electronic pressure transducers by detecting the pressure through the filter medium under testing. The test was conducted at room temperature (22 ± 3°C) with a flow rate of 85 ± 3 L/min. The air permeability was measured by a Frazier Air Permeability Tester (FX3300; Textest AG, Switzerland) with an effective area of 20 cm$^2$ and a pressure drop of 200 Pa. The water vapor transportation rate of the relevant membranes was investigated by the computer-type fabric moisture permeability testing apparatus (YG601H-II; Ningbo Textile Instrument Factory, China) based on the GB/T 12704.2-2009 standard test method.

3 Results and discussion

3.1 Morphology

In order to obtain a high-performance filtration membrane with both good breathable and directional moisture transport properties for the face mask that has favorable wearing comfortability, an asymmetrically superwettable membrane with a surface wettability gradient was constructed by integrating superwettetable skin layers with an open porous structured composite membrane. The basic composite fibrous membrane with nonwoven architecture was obtained by the versatile, readily accessible electrospinning technique. The representative SEM images of the PAN NFMs derived from various solution concentrations and the PEI NFMs shown in Figure 2a–e illustrate that the electrospun PAN and PEI NFMs were 3D nonwoven membranes assembled with randomly oriented nanofibers. The average diameter of PAN nanofibers increased gradually from 211 to 715 nm as the solution concentration increased from 10 to 16 wt%. This should be attributed to the enhanced viscosity and surface tension extensively studied in the previous studies (15–17). The open network porous structure changed accordingly as the fiber diameter varied. The PSD also exhibited the same increasing trend with the changes in the fiber diameter as displayed in Figure 2g. Besides, the mechanical robustness, an important parameter in practical face masks, is significantly affected by the solution concentration (18). The tensile stresses of PAN NFMs and PEI membranes are 2.47, 2.89, 3.36, 4.42, and 10.65 MPa, respectively. All the NFMs show a characteristic nonlinear elongation behavior until breakage, which is ascribed to the dynamic deformation and the alleged “pull-out” slipping of single fiber along the stress loading direction. The frictional entanglement resulting from the increase in the fiber diameter and the slipping resistance may be the main reasons for the incremental tensile stress (19,20).

On the basis of the fact that the fiber diameter and pore structure would have a great influence on the capture process of PM$_{2.5}$, the air filtration performance of
the obtained PAN/PEI composite membranes with various microstructures was carefully studied. Figure 3a presents the dependence of the filtration efficiency of the PAN/PEI composite NFMs on the particle size in the range of 0.2–7 µm. The filtration efficiency of NFM10 (93.6%) is superior compared with that of NFM16 (80.6%) toward 0.3 µm particles at a basis weight of 0.6 g m$^{-2}$. Meanwhile, the filtration efficiency of NFMs improved with the increase in the particle size, owing to the effective interception mechanism. Besides, the filtration performance strongly relies on the packing density (21). The low packing density of membrane would reduce the tortuous path for the air flow to travel through, thus reducing the pressure drop of NFM. Although the samples from NFM10 to NFM16 have the equivalent basis weight, they displayed different pressure drops resulting from the change in the packing density (Figure 3b). The trade-off parameter between the pressure drop and filtration efficiency, known as quality factor (QF), was used to evaluate the overall filtration performance (22,23). The QF was calculated by the formula as follows:

$$QF = -\ln(1 - \eta) \frac{\Delta P}{\text{DP}},$$

where $\eta$ is the filtration efficiency (%) and DP is the pressure drop (Pa) of the relevant NFM. As shown in Figure 3b, the QF values are 0.059, 0.0639, 0.0637, and 0.0672 Pa$^{-1}$ for the bilayer NFMs, respectively, indicating the benefit-to-cost roles of fiber and pore structure toward the construction of bilayer structure.

### 3.2 Performances of PAN/PEI membranes

According to the previous studies, air permeability and moisture transfer are two important indicators of the wear comfort behavior for human respiration (9). Figure 3c and d shows the variations in air permeability and
WVTR with the solution concentration of PAN. The air permeability improved from 147 to 385 mm s\(^{-1}\) with the increase in the PAN concentration, and the values present positive correlation with pore size, as shown in Figure 3c. It is obvious that a forward WVTR of 1.56 kg m\(^{-2}\) d\(^{-1}\) and a ΔWVTR of 0.62 kg m\(^{-2}\) d\(^{-1}\) were achieved when the PAN concentration was 10 wt%, suggesting the good moisture transfer performance of the NFM10 was due to the relatively higher porosity.

3.3 Morphology of skin layers

In order to improve the directional moisture transport ability, a functional skin layer with asymmetric superwettability was fabricated via a successive electrospraying method to obtain a surface energy gradient through the membrane. Figure 4a and b displays the typical SEM images of the optimal hydrophilic and superhydrophobic surfaces. It can be observed that all the microspheres with a diameter ranging from 0.2 to 1.8 \(\mu\)m hang together with the ultrathin fibers. The SiO\(_2\) NPs played a key role in constructing nanoscale roughness on the surfaces of microspheres and fibers, because the polymer droplets would shrink during the solvent evaporation process (24,25).

As can be seen in Figure 4c, an irregular pore structure is confirmed for the bilayer PAN/PEI NFM with a pore size in the range of 1.26–1.61 \(\mu\)m (average pore size: 1.44 \(\mu\)m), and this is due to the different diameters of PAN and PEI fibers. Nevertheless, after spraying the SiO\(_2\)@PAN and F-SiO\(_2\)@PEI solutions onto the NFM substrate, we observed the substantial decrease in the average pore size to 1.27 \(\mu\)m, which is attributed to the dense accumulation of microspheres-on-string-structured fibers.

Figure 4d shows the water contact angle (WCA) of the corresponding layer of the composite membrane, and the WCA values of PAN, PEI, SiO\(_2\)@PAN, and F-SiO\(_2\)@PEI are 76.6°, 132.1°, 26.3°, and 153.7°, respectively. The asymmetric wettability was obtained to investigate the effects of hydrophilic degree and surface roughness on the directional moisture transport process. Additionally, the skin layers strongly adhered to the bilayer PAN/PEI NFM substrate, resulting from the solvent that did not evaporate in time and the robust electrostatic forces produced in the electrospraying process.

Figure 3: Filtration efficiency (a), pressure drop (b), air permeability and QF (c), and WVTR (d) of PAN/PEI composite membranes with varying PAN concentrations. The basis weight of the composite NFMs was 0.6 g m\(^{-2}\).
3.4 Filtration performances of multilayered composite membranes

The integration of skin layer endowed the membranes with a desirable surface roughness, small fiber diameter, and high effective surface area, which greatly reduced the pore size and fiber contact area, finally enhancing the filtration performance. The pore sizes of all samples ranged from 0.44 to 2.37 µm and sequentially declined from 1.92 to 1.25 µm with the increase in the fiber basis weight from 3 to 10.5 g m$^{-2}$ (Figure 5a). The pore size was inversely proportional to the fiber basis weight, owing to the gradual reduction in the tortuosity and pore interconnectivity of the fibrous membranes.

Figure 5b displays the dependence of the filtration efficiency of the SNFMs on the 0.2 to 2.5 µm particles. The results showed that the increased fiber mass was likely to result in the increase in the contact points in the fibrous media and the tortuous airflow channels, finally leading to the improved pressure drop (64–338 Pa) (Figure 5c). Interestingly, the particle capture efficiency of SNFMs at a small fiber basis weight was much lower than that of large particles (larger than 0.3 µm). This phenomenon can be explained by the straining of aerosols as well as the diffusion and interception mechanisms when the size of ultrafine particles is close to the fiber diameter (21,26).

Figure 5d displays the QF values of various fiber media as a function of basis weight. The commercial polypropylene (PP) melt-blown fibers possess a relatively high QF value of 0.5862 Pa$^{-1}$, but have a large basis weight (>90 g m$^{-2}$), which are not beneficial for breathing. The lightweight character of the electrospun fiber-based media (<10 g m$^{-2}$) can not only promote the wearing comfort performance but also give more chance for the entire design of the protection masks. Besides, benefiting from the multivariate fiber structures and construction processes, the QF can be further increased, on account of the enhanced capture efficiency. A high QF value of 0.1089 Pa$^{-1}$ was achieved for the SNFM fibrous media with a basis weight of 3 g m$^{-2}$ (Figure 5d), much higher than that for the previously reported electrospun media (27).

3.5 Wearing comfortability

To evaluate the wearing comfortability of the as-prepared composite SNFMs, air permeability and moisture transfer performance were systematically investigated (28). The variations in air permeability and ΔWVTR with various
fiber basis weights are illustrated in Figure 6a. Obviously, the air permeability value was significantly reduced from 278 to 21 mm s\(^{-1}\) with the increase in the fiber mass, which could be explained using the Fickian diffusion model. Specifically, the diffusion flux is positively correlated with the diffusion coefficient that is indirectly determined by the fiber basis weight (29). Interestingly, a ΔWVTR value of 3.61 kg m\(^{-2}\) d\(^{-1}\) was obtained when the fiber mass was fixed at 3 g m\(^{-2}\), demonstrating the excellent unidirectional moisture transport property of the sample of SNFM3. Based on this, a conclusion may be drawn that the wearing comfortability can be effectively regulated by controlling the fiber basis weight.

In addition, there is an urgent need for the thermal comfort face masks that could greatly save the energy consumption of human body, especially in high-temperature and high-humidity environments (30,31). The thermal infrared transmittance of SNFM3 and the commercial PP membrane were 92.1 and 2.16%, which indicated that the as-prepared composite medium possessed a distinct radiative cooling property (Figure 6b). This result could be interpreted as almost all body radiation was transmitted by the ultralight mass and high porosity (32,33). The commercial face mask with a large pore size (>1 μm) and high basis weight (>90 g m\(^{-2}\)) usually traps air and creates insulation inside the pores to block heat transfer, thus leading to low radiation capability. The relevant comparison of the relevant properties of the multilayer-structured SNFM3 and commercial PP membrane is displayed in Table A1.

4 Conclusions

In summary, we have demonstrated a feasible and facile strategy to develop nanofibrous filter media for the face masks with excellent properties. By tailoring the structures, pores, and wettability of the composite membranes, the filtration performance and wearing comfortability were greatly improved. It has been proved that the air and moisture permeability was linearly dependent on the porosity. Moreover, because of the ultralight basis weight and across-thickness wettability gradient, the SNFM3 exhibited a high filtration efficiency (99.3%), a low pressure drop (64 Pa), a good air permeability (278 mm s\(^{-1}\)), and a desirable water vapor permeability difference (3.61 kg m\(^{-2}\) d\(^{-1}\)), which are beneficial to the actual applications in personal protective
devices. Importantly, the SNFM3 presented a stable radiative dissipation ability, capable of maintaining the face cool and comfortable in a hygrothermal environment. This work could provide a versatile method for designing multifunctional fibrous media for high-performance face masks. More significantly, how to promote the effective utilization in a high-moisture environment with wearing a face mask is still a challenge. Electrical stimulation motivated by moisture can not only obtain lots of electric charges to capture air pollutants but also achieve the antibacterial property; this will be an attractive development direction in air filtration.

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## Appendix

Table A1: Comparison of the relevant properties of multilayer structured SNFM3 and commercial PP membranes

| Sample        | Basis weight (g m⁻²) | Air permeability (mm s⁻¹) | Comparable water vapor permeability (kg m⁻² d⁻¹) | Filtration efficiency (%) | Pressure drop (Pa) | FTIR transmittance (%) |
|---------------|----------------------|----------------------------|----------------------------------------------------|---------------------------|-------------------|------------------------|
| SNFM3         | 3.0                  | 278                        | 3.61                                               | 99.3                      | 64                | 92.1                   |
| PP membrane   | 90.2                 | 203                        | 0.48                                               | 99.7                      | 132               | 2.16                   |