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PAPER

Electrospinning fabrication of polystyrene-silica hybrid fibrous membrane for high-efficiency air filtration

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Abstract

The development of new materials for air filtration and particulate matter (PM) pollution is critical to solving global environmental issues that threaten human health and accelerate the greenhouse effect. In this study, a novel electrospun polystyrene-SiO₂ nanoparticle (PS-SNP) fibrous membrane was explored by a single-step strategy to obtain the composite multi-layered filter masks. In addition, the air filtration performance of this fibrous membrane for PM was evaluated. The effects of SiO₂ on the composition, morphology, mechanical property, and surface wetting of PS-SNP membranes were studied. Allowing SiO₂ to be incorporated into the PS polymer was endowed with promising superhydrophobicity and demonstrated excellent mechanical properties. As-prepared PS-SNP membranes possess significantly better filtration efficiency than pure PS membrane. Furthermore, a three-layered air filter media (viscose/PS-SNP/polyethylene terephthalate) used in this study has considerable performances compared to the commercial masks. Since this air filtration membrane has excellent features such as high air filtration and permeability, we anticipate it to have huge potential application in air filtration systems, including cleanroom, respirator, and protective clothing.

1. Introduction

In the past ten years, the problem of air pollution is becoming more and more serious. Many pollutants such as PM2.5 and PM10 are produced by coal use and combustion of fossil fuel [1]. Most areas of industrial cities and their surroundings often appear of fog and haze weather, and in the short term, it is difficult to be improved obviously. Particles represented by PM2.5 can enter alveoli and blood, which causes irreversible effects on human health [2–5]. As people's awareness of self-protection is gradually increased, all kinds of anti-haze respirators are emerging. Besides, a recent outbreak of novel coronavirus pneumonia has spread rapidly around the globe [6–8]. The World Health Organization (WHO) recommends using masks as a set of public health interventions to prevent and control the spread of certain viral respiratory diseases [9]. Therefore, the design and research of high-efficiency filtration membrane used in the mask or other filtration devices is a central focus.

It is well known that electrospinning is an effective and environmentally friendly method that is widely used in producing continuous fiber membrane fields [10–13]. The prepared membrane exhibits controllable fiber diameter and controllable porous structure, making it an ideal choice for air filtration materials. According to these characteristics, many materials used for electrospun fiber membranes, such as polyurethane (PU), poly (acrylic acid) (PAA), polyethyleneimine (PEI), polyvinyl acetate (PVA), and polyacrylonitrile (PAN), have been reported [13–17]. Polystyrene (PS), a kind of colorless thermoplastic plastic, has a wide range of applications in the field of chemistry, life sciences, and medical treatment when it is prepared into fibrous membranes [18–21]. Electrospun fibers usually have a relatively smooth surface, which greatly influences the filtration properties of membranes. Allowing nano-inorganic materials to be incorporated into fibers membrane has been a widelyused method to construct hybrid structures and increase materials roughness. There are three methods to integrate inorganic materials into fibrous membrane: (i) 'pre-add' mixing nanomaterials to a spinning solution before electrospinning [22]; (ii) 'after-grow' in situ synthesis nanomaterials after membrane electrospinning [23]; (iii) 'multi-jet' electrospraying of nanomaterial dispersions and simultaneous electrospinning of polymer solutions [24]. Hybrid fibrous membranes with inorganic materials using the electrospinning method exhibit good filtration efficiency. For example, Q Ke et al prepared hierarchically structured TiO₂/PAN fibrous membranes, which showed excellent filtration efficiency and toluene degradation performance [25]. G Singh et al reported a polyvinylidene fluoride (PVDF)-Ag-Al₂O₃ nanofibrous membrane, which exhibited not only excellent filtration efficiency but antibacterial and detoxifying properties [26]. Moreover, S Wong et al studied that a critical loading of nano-scale graphite platelets (NGP) within PVDF membranes could improve both filtration efficiency and pressure drop [27]. Besides, due to the excellent electret performance created by dipole orientation and charge storage, SiO₂ is considered a promising material for designing new fibrous materials for air filtration [28–30]. It could enhance the mechanical properties and hydrophobicity of polymer fibrous membranes. It was reported that the SiO₂ nanoparticle was incorporated into PAN or PEI fiber membranes by pre-adding nanoparticles into a spinning solution to enhance the filtration performance [22, 31].

In this work, SiO₂ nanoparticles (SNP) were incorporated by the electrospinning technology to controllably manufacture hierarchical polystyrene (PS) fiber membrane with strong filtration performance for the first time. In addition, the combination of SNP has a great influence on the morphology, surface wettability, and mechanical property of the resulting PS-SNP membranes. More significantly, the PS-SNP fibrous membrane was assembled with viscose and polyethylene terephthalate (PET) into a three-layered air filtration media, which exhibits comparable performances of air filterability and permeability compared with other mask products sold on the market.

2. Experiments

2.1. Materials

The polystyrene (PS, Mw = 192000) was obtained from Sigma-Aldrich. The N, N-Dimethylformamide (DMF) was of analytical grade and supplied by Xilong Chemical Co., Ltd SiO₂ nanoparticles (with a diameter of 100 nm) were purchased from Beijing Chemical Reagents Co., Ltd. All reagents are used as received, no further purification is required.

2.2. Polymer solutions preparation

A PS solution (20 wt.%) was prepared by dissolving in DMF 24 h under stirring. SiO₂ nanoparticles (SNP) were then dispersed to this solution to prepare PS/SNP fibrous membranes. Additionally, PS solutions containing 0, 2, 5, 8, 10, and 12 wt.% SNP were prepared, respectively, using a DMF mixture.

2.3. Fabrication of PS/SNP fibrous membrane

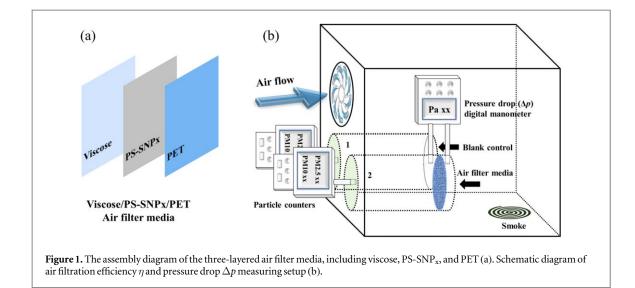
The PS solution was loaded into a jet and injected through a metal needle at a feed rate of 3.0 ml h⁻¹. Since a high voltage of 20 kV is applied to the needle tip, a continuous jet is generated. The PS fiber membrane was deposited on aluminium foil 20 cm from the tip of the syringe. Then, the obtained electrospun membrane was dried in a vacuum for 2 h to remove residual solvent. The PS/SNP hybrid fibrous membranes containing various contents of SNPs were denoted as PS-SNP_x (where x is the mass ratio of SiO₂ nanoparticles in hybrid membranes).

2.4. Characterization

The PS and PS-SNP_x membranes' morphologies were characterized using a scanning electron microscope (SEM, JSM7500, JEOL, Tokyo, Japan). The water contact angle measurements were performed using a water contact angle goniometer (JC2000C, Powereach Co., Shanghai, China). The mechanical properties were tested on a tensile tester (XQ-1C, Shanghai New Fiber Instrument Co., Ltd, China) at a crosshead speed of 5 mm per minute at room temperature. X-ray photoelectron spectroscopy (XPS) spectra were conducted using a VSW spectrometer equipped with a monochromatized X-ray source (Al K α $h\nu$ = 1486.6 eV). The take-off angle was 90°, and the energetic resolution was 0.2 eV. Data analysis was performed with the CasaXPS software.

2.5. Air permeability and filtration performance

 $PS-SNP_x$ electrospun fibrous membrane was assembled with viscose and polyethylene terephthalate (PET) into a three-layered structure (figure 1(a)). Viscose and PET non-woven fabric are the most commonly used materials for masks due to the physiological moisture requirement to human skin and having relatively good air



permeability. In this study, viscose was used as the inner layer, PET was used as the outer layer, our PS-SNP fibrous membranes were used as the middle layer. Therefore, the front and back sides of the PS-SNP fibrous membranes were covered with PET and viscose forming an air filter media. The edges of the three-layers film were fixed by nylon sewing thread to prevent interlayer peeling. An assembled sandwich-type air filter media (viscose/PS-SNP_x/PET) was made to compare with the other sold masks on the market.

The air filtration and the pressure drop were performed in a homemade system (figure 1(b)). It shows a test setup composed of two chambers at the bottom of a container ($25 \text{ cm} \times 35 \text{ cm} \times 50 \text{ cm}$). Chamber 1 is a cylindrical shaped structure (internal diameter 12.6 cm) with a hole (3 cm in diameter) at the left side for the particle counter. The right side is completely open to the container. Chamber 2 is identical to chamber 1. However, on the right side, the air filter media (viscose/PS-SNP_x/PET) was well installed in the cylindrical shaped structure by tapes during the study to separate chamber 2 from the container. Hence, chamber 1 is a blank control structure compared with chamber 2. In addition, a smoke generator is located inside the container on the right side. Meanwhile, a blower is used to induce the airflow into the two processing chambers. In this work, the smoke (considered as the source of PM) was produced by burning a cigarette. Its smoke was firstly isolated in the container and then inhaled in all subsequent chambers during the filtration process. PM concentration was measured by particle counters (in μ g m⁻³, test X in HT9600) from chamber 1 (blank control) and chamber 2 (after air filter media), respectively. The environmental conditions were maintained at constants: 25 °C with a relative humidity of 60%. The air filtration efficiency (η) was calculated using the equation (1) [12]

$$\eta = 1 - \frac{C_{\text{filter}}}{C_{\text{blank}}} \tag{1}$$

where C_{blank} is the concentration of PM2.5 taken from the blank control chamber 1 and C_{filter} is that taken from chamber 2 after the air filter media. The pressure drop (Δp) was obtained by a digital manometer measuring the pressure difference across the sample (blue, marked in figure 1(b)) during filtration tests. To determine the overall performance of this air filter, the quality factor (QF) was calculated by the experimental data of filtration efficiency and pressure drop using the equation (2) [12]

$$QF = -\frac{\ln(1-\eta)}{\Delta p}$$
(2)

3. Results and discussion

SEM images of the PS-SNP_x with different amounts of SiO₂ nanoparticles are shown in figure 2. As observed, the prepared membranes exhibit an interconnected porous structure, and the fibers are randomly oriented. Figure 2(a) shows the very smooth surface morphology of pure PS fibers. However, after introducing SiO₂ nanoparticles (figures 2(b)–(f)), granular particles are dispersed on the surface of PS-SNP_x fibers, which indicates that the SiO₂ nanoparticles have been successfully doped into the membrane. The PS-SNP_x membranes contain hybrid fibers with an average diameter of approximately 2.5 μ m (figure S1, see supporting information (available online at stacks.iop.org/NANOX/2/020017/mmedia)). It is obvious that the fiber diameters of different membranes are not much different, which indicates that the introduction of nanoparticles does not significantly change the diameter of the electrospun membranes. On the contrary, with the increase of

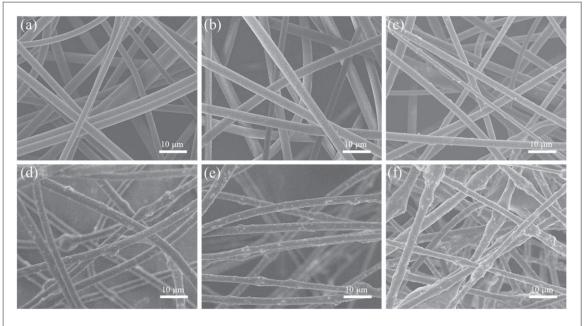
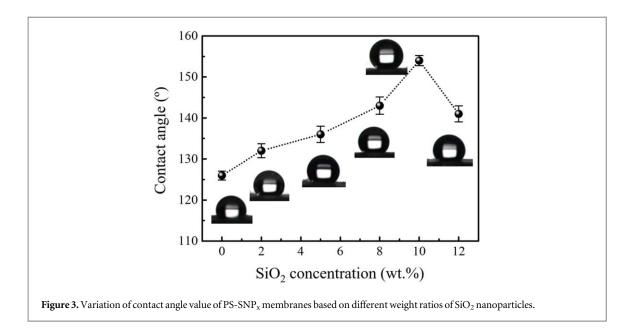
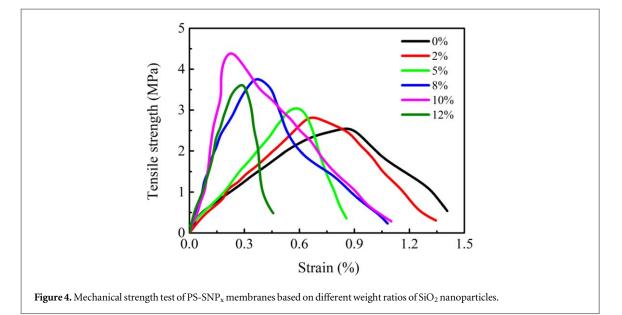


Figure 2. SEM images of PS-SNP_x under different SiO₂ nanoparticles concentrations. (a) 0 wt.%; (b) 2 wt.%; (c) 5 wt.%; (d) 8 wt.%; (e) 10 wt.%; and (f) 12 wt.%.



SiO₂ content, the morphology of the fiber has been changed considerably. The more nano-scale protrusions were formed on the surface, the rougher the morphology of membranes became. It can be concluded that this work provides a simple method to construct structured membranes in which SiO₂ nanoparticles are distributed on fibers controlled by different amounts.

Figure 3 displays the water contact angle (CA) value of the PS-SNP membranes as a function of SiO₂ nanoparticle concentration. The wettability of the composite fiber membrane decreases with the increasing amount of SiO₂ nanoparticle from 0 to 10 wt.%. The CA increases from 126° to 155° , indicating a superhydrophobic surface. However, the CA of the fiber membrane drops to 140° when the amount of SiO₂ nanoparticle up to 12 wt.%, which is mainly based on the alternation of surface morphology. Initially, the pure PS fibers have very smooth surfaces (figure 1(a)). When SiO₂ is involved (2 wt.%), the nanoparticle attachment appears on the surface of the fibers, which begins to raise the roughness of PS-SNP membranes, so that the CA is improved. Continue to increase the SiO₂ concentration up to 10 wt.%, SiO₂ adhesion rate expands significantly. Simultaneously, a large number of grooves and edges appear, which amplifies the roughness of the film surface. Further increasing the SiO₂ concentration (12 wt.%), the CA value is decreased to 140° , probably resulting from that SiO₂ agglomeration phenomenon destroy the dual-scale structure and trap less air in surface cavities [32].



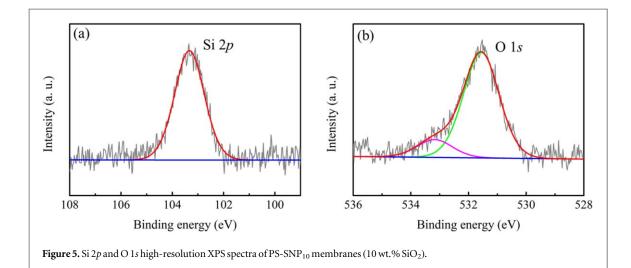
Besides, the introduction of hydrophilic SiO₂ nanoparticles affects the hydrophobicity of the PS-SNP membrane surface.

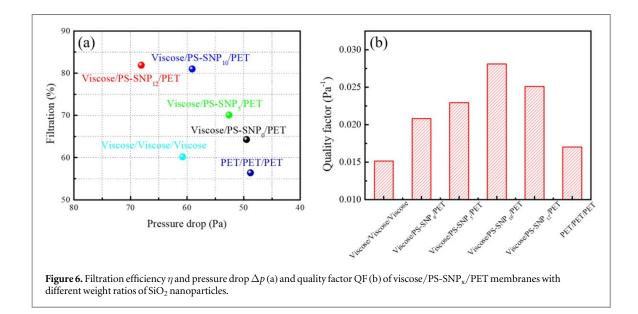
The tensile properties of pure PS and PS-SNP_x membranes are shown in figure 4, where the maximum tensile strength variation and the stress-strain diagram are plotted. The mechanical properties values of the pure PS and PS-SNP_x are given in table S1 (see supporting information). After adding SiO₂ nanoparticles, the tensile strength of the membrane is improved. The difference in each membrane strength-strain curve is mainly caused by SiO₂ content. Electrospun pure PS membranes are generally soft and flexible, with a tensile strength of less than 2.55 MPa. It can be seen that the increase of tensile strength is related to the presence of SiO_2 . Compared to pure PS membranes, the tensile strength of PS-SNP $_{10}$ composites is enhanced by 72% from 2.55 to 4.39 MPa. This improvement in mechanical properties is evidence of the transfer of efficient load to SiO_2 in PS-SNP_x membranes. The enhancement of the mechanical property of PS-SNP_x membranes has probably resulted from the strong interaction between PS and SiO₂. Firstly, SiO₂ acts as a cross-linking point to associate with polymer chains, thereby increasing the rigidity. Secondly, the inorganic nanoparticles on the composite electrospun fiber rises the friction between adjacent fibers [33, 34]. Besides, the tensile strength of the membrane is slightly decreased at 12 wt.% SiO₂ content. This decrease could be attributed to the uneven distribution of SiO₂ in the polymer matrix, which leads to an aggregation of clay. These aggregates cause the stress to be concentrated at various points in the polymer, thereby reducing the mechanical properties. However, it is still higher than that of the pure PS membrane. The positive effect of SiO_2 depends largely on the dispersion of SiO_2 nanoparticles in the matrix and its interface interactions [35].

Apart from tensile mechanical properties, the bending ability is also a notable point in membrane or film materials [36, 37]. Because this is very important for the application of flexible devices and commodities. In this study, both PS and PS-SNP_x membranes exhibit good flexibility during materials characterization and filtration tests. Figure S2 (see supporting information) shows an optical image of the fold PS-SNP₁₀ membrane. Although induced by SiO₂, the PS-SNP membrane was flexible and no obvious cracks appeared. Additionally, as another determination of the flexibility, the bending rigidity test will also be considered in the follow-up work to provide more comprehensive proof.

X-ray photoelectron spectroscopy (XPS) is used to investigate surface compositions of the PS-SNP₁₀ membranes when the content of SiO₂ is 10 wt.%. For the pure PS membrane, the Si 2*p* and O 1*s* peaks were not detected as expected. However, they appear obviously on PS-SNP₁₀ membranes surface, as shown in figure 5. From the high-resolution Si 2*p* spectrum (figure 5(a)), the signal (103.5 eV) is due to the Si-O bond. The O 1*s* high-resolution spectrum shows two visible signals, the peak at a binding energy of 531.5 eV was related to the Si–O bond; another at a binding energy of about 533.3 eV was probably associated with the surface oxygen contamination (figure 5(b)) [38]. The XPS results indicate the incorporation of SiO₂ nanoparticles into the PS.

The filtration performance of the viscose/PS-SNP_x/PET air filter media with various concentrations of SiO₂ nanoparticle is demonstrated in figure 6(a). It is worth noting that the PS-SNP_x (x = 0, 5, 10, and 12 wt.%) membranes were assembled with viscose and polyethylene terephthalate (PET). In addition, the viscose/viscose/viscose and PET/PET/PET three-layered filter media were also prepared and compared as control. As shown in figure 6(a), the abscissa is the pressure drop (Δp), and the ordinate is the filtration efficiency (η). The filtration efficiency of viscose/PS-SNP_x/PET membranes versus the increasing SiO₂ content of 0, 5, 10, and 12





wt.% are 64.3%, 70.1%, 81%, and 81.9% respectively, while the corresponding pressure drop are 49.5, 52.6, 59.1, and 68.1 Pa respectively. In this case, as more SiO₂ nanoparticles are incorporated into the PS-SNP, the values of both Δp and η gradually increase. Furthermore, although the viscose/PS-SNP₁₂/PET shows the best efficiency ($\eta = 81.9\%$), the Δp value is the highest among all samples, revealing the worst air permeability. The η of viscose/PS-SNP₁₀/PET is about 81%, slightly lower than that of viscose/PS-SNP₁₂/PET. However, its Δp value is about 59.1 Pa, which is much lower than that of viscose/PS-SNP₁₂/PET (68.1 Pa). Generally, a compromise needs to be achieved between these two important filtration-permeability parameters: high η and low Δp . To evaluate the overall filtration performance of the membranes, the quality factor (QF) is further calculated in figure 6(b), which also takes into account both the efficiency and the permeability. The results show that since the SiO₂ concentration is increased from 0 to 10 wt.%, the QF value is then improved from 0.0208 to $0.0281 \, Pa^{-1}$. However, when the concentration rises to $12 \, wt.\%$, the QF value begins to decrease slightly (0.0251Pa⁻¹). Besides, the two control samples (viscose/viscose/viscose and PET/PET/PET) exhibit relatively lower QF values, indicating the insufficient balance between breathability and filtration. Overall, the viscose/PS-SNP_x/PET air filtration media has the best performance at 10 wt.% SiO₂ doping. It shows that this viscose/PS-SNP₁₀/PET has good comprehensive filtration performance and great application prospects in the purification of PM2.5.

SiO₂ is considered a promising candidate for the design of new fibrous materials for air filtration because of excellent electret behavior, which can retain electric charges over a long period of time and create an external electric field [39, 40]. Furthermore, in the study of electrospinning fiber membranes, the introduction of SiO₂ provides the surface with a rougher structure, superhydrophobic property, and changes in fiber diameter and pore size [22, 23]. According to previous studies, inertial impaction, interception, and diffusion are the most important filtration mechanisms for air filtration according to classical filtration theory [41–43]. In this work,

the enhanced filtration performance could be attributed to the spatial-constructing ability of SiO₂ nanoparticles. During the filtration, a stagnant area will be formed in the boundary layer around the nano-protrusions modified rough fibers [31, 44, 45]. The fine particles move slowly like a peristaltic flow and stay at the edge of fibers. The rough structure improves the filtration efficiency and promotes air penetration through the membrane, which greatly improves the filtration performance. Additionally, the SiO₂ electret effect may be important to charge trapping and enhance the filtration properties [22, 29].

The filtration performance of commercial masks was also studied for the effective follow-up comparison with PS-SNP_x fiber membranes. The experimental results are shown in table S2 and plotted in figure S3 (see supporting information). The QF of the viscose/PS-SNP₁₀/PET air filtration media has a higher value than the commercial three-layered masks. Then the long-term filtration performance of air filtration was evaluated because it is significant for practical applications. As shown in figure S4 (see supporting information), after 8 h, the viscose/PS-SNP₁₀/PET sample retains 92.1% of its initial filtration efficiency, showing long-term high stability, which is still higher than that of the tested commercial facial masks.

To the best of our knowledge, in addition to the air permeability property, the water-vapor permeability is also an important indicator of the parameters of membrane masks [46]. The water-vapor transmission rate (WVTR) could be measured and determined by the water-proof breathability [47]. As a result, the water-proof-breathable membrane is a kind of function material that can prevent the penetration of liquid water but allows water vapor/air to pass through, and it is widely used in protective clothing, chemical industries, and medical equipment [48]. It is reported that the electrospun fibrous membranes had good water-proof breathability [49, 50]. Besides, by introducing inorganic nano-SiO₂ to change the pore structure and hydrophobicity of the electrospun membranes, many studies have been carried out to enhance this performance [51–53]. The next step of this study will consist of performing the development and improvement of the water-vapor permeability of the novel PS-SNP fibrous membranes.

4. Conclusion

In summary, we developed novel and simple PS-SNP fibrous membranes utilizing electrospun PS fibers and SiO₂ nanoparticles for effective air filtration. The use of SiO₂ nanoparticles gives the fiber membrane superhydrophobicity and a relatively increased mechanical behavior compared with the pure PS membranes. More significantly, a composite PS-SNP fibrous membrane is assembled with the viscose and PET to be achieved a three-layered mask material. The as-prepared air filtration membrane has good air filtration, permeability performance and provides a general strategy for continuously exploring high-performance fiber media. It is concluded that the proposed PS-SNP air filtration membranes have high performance for filtering fine particulate matter. This fiber membrane-based mask provides new possibilities and concepts for existing masks on the market.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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