



Hierarchical Porous Recycled PET Nanofibers for High-Efficiency Aerosols and Virus Capturing

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Hierarchical ~~porous recycled-PET nano-fibers~~ Porous Recycled PET Nanofibers for ~~high efficiency aerosols~~ High-Efficiency Aerosols and ~~virus capturing~~ Virus Capturing

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29-09-2021

Abstract

Plastic crisis, especially for ~~polyethylene terephthalate~~ poly(ethylene terephthalate) (PET) bottles, has been one of the greatest challenges for the earth and human beings. Processing ~~recycled-PET~~ recycled PET (rPET) into functional materials has the dual significance of both sustainable development and economy. Providing more possibilities for the engineered application of rPET, porous PET fibers can further enhance the high specific surface area of electrospun membranes. ~~Here~~ Here, we use a two-step strategy of electrospinning and ~~post-processing~~ postprocessing to successfully control the surface morphology of rPET fibers. Through a series of optical and thermal characterizations, the porous morphology formation mechanism and crystallinity induced by solvents of rPET fibers ~~was~~ were discussed. Then, this work further investigated both PM2.5 air pollutants and protein filtration performance of rPET fibrous membrane. The high capture capability of rPET membrane demonstrated its potential application as an integrated ~~high-efficiency~~ high-efficiency aerosol filtering solution.

Keywords

waste bottle; electrospinning; porous fiber; ~~polyethylene terephthalate~~ poly(ethylene terephthalate) (PET); air filtration

1. Introduction

Graphic abstract
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Declaration of Competing Interest
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
Data Availability
Table of Contents (TOC)
The United Nations sustainable development goals are committed to being carbon neutral on earth.¹ As the major carbon emission states, China, the European ~~Union~~ Union, and the United States ~~are~~ have proposed their roadmaps ~~toward~~ toward more sustainable economies.² Food and beverage packaging is an indispensable part of modern lifestyle and economies. However, the current disposal of beverage plastic bottles made from PET has been a serious concern for decades due to their massive pollution on the environment and human beings, though an alternative sustainable roadmap is promoting biodegradable plastic bottles and packages.^{3,4} Furthermore, the demand for personal ~~protect~~ protective equipment (PPE, including protective suit, face ~~masks;~~ mask, face shield), which is superimposed on the global spread of the COVID-19 epidemic, remains high.⁵ The supply chain and sustainable recycling of polymeric plastic raw materials have become urgent problems in the very near future.

Recycled PET (rPET) has been widely used in the textile industry to produce ~~non-woven~~ nonwoven fabrics, garments, ~~ropes~~ ropes, and other chemical engineering materials. Therefore, the investigation of recycled engineering is ~~not only~~ a solution not only to the sustainable ~~development;~~ development but also for improving the value ~~to~~ of PET products.

Electrospinning is a facile technology to produce fibers with ~~nano/micro—scales~~ [nano/microscales](#) in quantity. Although the ~~non-woven~~ [nonwoven](#) membranes stacked by the electrospun fibers have a large number of voids and holes, these fibrous membranes could not be regarded as porous electrospun fibers.⁶ Porous fibers should refer to the pits or voids on the surface or interior of the individual single fiber. Combining with the holes on electrospun nonwoven membranes, such materials can be considered as hierarchical porous fibers. Hierarchical porous electrospun fibers show advantages in a variety of applications including tissue engineering,⁷ protective and smart ~~clothing~~ [clothing](#), and filtration due to their higher specific surface area and roughness interfaces.^{8,9} For ~~instances~~, [instance](#), hierarchical porous fibrous membranes have more contact area to intercept pollutant particles in gas medium and to load more pollutants.^{10,11} The ~~micro-hierarchy~~ [microhierarchy](#) structures can also mimic human extracellular matrix and elevate cell attachment and proliferation performances.¹²

~~Conventional~~ [The conventional](#) porous formation mechanism for electrospinning process is similar to the template casting fabrication.¹³ The formation of pores during electrospinning is essentially a process of controlling the solidification of the polymer from the solution. According to the strategy of the curing process, the porous formation mechanisms at this stage can be divided into ~~thermal-induced~~ [thermal-induced](#) phase separation (TIPS) that ~~involving~~ [involves](#) temperature ~~changing,~~ [solvent-induced changes](#), [solvent-induced](#) phase separation (SIPS) that selects a highly volatile solvent, or ~~non-solvent-induced~~ [non-solvent-induced](#) phase separation (NIPS) that applies diffusive exchange with a ~~non-solvent~~ [nonsolvent](#) during spinning or casting.¹⁴ Specifically, most ~~of~~ researchers have generally accepted in recent years that the trifluoroacetic acid (~~TFA~~)/ ~~dichloromethane~~ (~~TFA~~)/ [dichloromethane](#) (DCM) solvent system is the mainstream choice for electrospinning PET fibers though the specific solution ratios were different.^{15–17} However, this choice can only prepare conventional smooth fibers. As a case of SIPS, Shu et al. reported PET fibers with some pits on the surface by applying hexafluoro isopropanol (HFIP)/ DCM solution systems under a ~~high humidity (60%–80%)~~ [high-humidity \(60–80%\)](#) environment.¹⁸ The precise and universal parameters for fabricating porous rPET fibers are worthy to be studied. Previous publications have repeatedly proved that these ~~of~~ pores formed during electrospinning are often ~~unform~~ [uniformly](#) distributed, and the improvement of the ~~fiber's~~ [fiber's](#) specific surface area is limited.

The majority of macromolecular electrospun fibers are in a low-crystalline or ~~non-crystalline~~ [noncrystalline](#) state.¹⁵ Therefore, organic solvent ~~system-induced re-crystallization~~ [system-induced recrystallization](#) of macromolecules is an alternative to modify ~~fiber's~~ [fiber's](#) hierarchical porous surface morphology after electrospinning. The ~~recent~~ [recently](#) published systematic research has successfully implemented this theory on PLLA fibers.¹⁹ rPET is a ~~co-polymer~~ [copolymer](#) that can ~~be induced re-crystallization~~ [induce recrystallization](#) by solvents.²⁰ In view of the dissolution characteristics of PET, the careful selection of solvent systems to swell polymers rather than dissolve them is the key to the successful fabrication of porous electrospun PET fibers. The selected solvent systems would enter [the](#) rPET fiber ~~interior~~ [interior](#), where the NIPS mechanism ~~formed~~ [takes place](#) during the electrospinning.

In this article, we used [a](#) conventional spinning solution at room environment that followed ~~by~~ a novel post-treatment strategy to prepare hierarchical porous rPET fibers. The crystalline and ~~the~~ porous surface of PET fibers can be controlled through adjusted parameters of [the](#) solvent system. The parameters of electrospinning preparation were ~~confirmed, while the~~ [confirmed during](#) post-treatment, i.e., a variety of solution systems for solvent-induced recrystallization have been designed and implemented. Subsequently, the surface morphology, solvent residue, thermophysics, and the crystallinity of rPET fibers were characterized. The mechanism of hierarchical porous formation was discussed on account of characterization data. Finally, the PM2.5 and virus filtration performances of electrospun rPET fibrous membrane were evaluated.

2. ~~Materials and~~ [methods](#) [Methods](#)

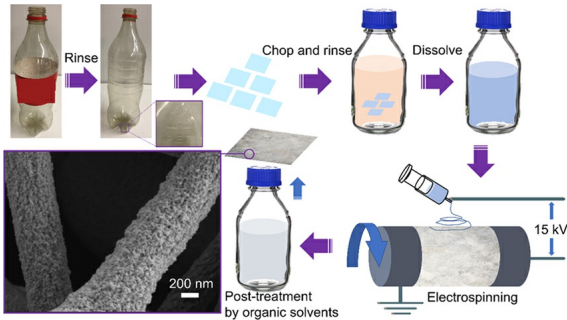
2.1. ~~Materials~~

The rPET used in this study was ~~Coca-Cola@Coca-Cola~~ coke plastic bottles ~~which~~ [that](#) were sold in [a](#) UK grocery. TFA, acetone, and ethanol were purchased from Alfa Aesar, ~~UK~~ [U.K.](#) DCM and ~~N-Methyl-2-pyrrolidone (NMP)~~ [N-methyl-2-pyrrolidone \(NMP\)](#) were purchased from ~~Sigma-Aldrich, USA~~ [Sigma-Aldrich](#).

2.2. ~~Electrospinning and~~ [post-treatment](#) [Post-Treatment](#)

~~Electrospun~~ [Electrospinning](#) and [the](#) consequent post-treatment ~~for~~ [process of](#) rPET ~~were~~ [are](#) demonstrated in Figure 1. rPET bottles were

shredded into small pieces about 5 × 5 mm² after rinsed with ethanol. PET pieces were dissolved by a dual-solvent system that was mixed by TFA and DCM by 1:1 ratio by weight. The PET electrospun solution (10% w/w) could be obtained after 24 h stirring at room temperature. TONG LI TECH Nanofiber Electrospinning Unit (Shenzhen, China) was used to produce rPET nano-fibrous nanofibrous membrane at 25 °C and 40% humidity. The PET/TFA/DCM spinning solution ejection feeding rate was 1 mL/h, while the receiving distance and supply voltage from electrospinning needle tip to drum collector were 25 cm and 15 kV, respectively. All sample groups were dried in a fume hood for 24 h before further processing to ensure the complete evaporation of organic solvents.



1. Schematic diagram of recycled PET electrospinning and post-treatment process.

The PET fibrous membranes were immersed in different organic solvents for 30 min as post-treatment. The specific design of experiments was shown in Table 1. For specimens after NMP/ethanol treatment, they were rinsed with ethanol absolute twice, 5 min each time to remove NMP solvent inside the fibrous membrane interior. All rPET specimens after solvent post-treatment were dried in the fume hood at room temperature overnight before further experimental analysis, to ensure the complete evaporation of the solvent in the samples.

1. Design of Experiments for electrospun rPET fibrous membranes

Sample ID	Post-treatment solvents (w/w)	Temperature (°C)
A	Acetone	25
E	Ethanol	25
N1	NMP/ethanol (50%:50%)	25
N2	NMP/ethanol (37.5%:62.5%)	25
N3	NMP/ethanol (25%:75%)	25
N4	NMP/ethanol (50%:50%)	-20

2.3. Characterizations

The surface morphology of PET fibers was observed by a field emission scanning electron microscope (FE-SEMs, Philips XL30, The Netherlands, and Zeiss Ultra55, Germany) after platinum coating.

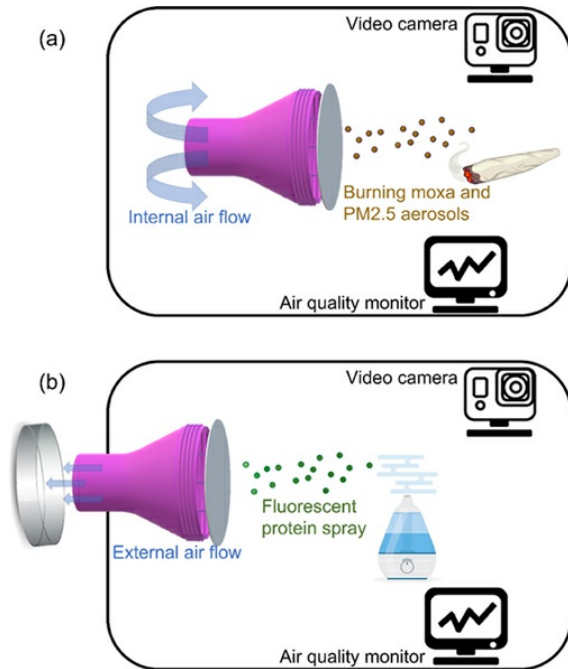
Fourier-transform infrared (FT-IR) spectroscopy was conducted by a Nicolet 5700 FT-IR Spectrometer (Thermo, USA) with an attenuated total reflectance (ATR) smart orbit attachment. Different scanning calorimetry (DSC) was performed using Q1000 (TA Instrument, USA) under a nitrogen (N₂) flow, with a heating rate of 8 °C/min from 30 to 300 °C. Each group of specimens has three samples. X-ray diffraction (XRD) was operated on a X'Pert Pro X-ray diffractometer (Panalytical, UK). The angle range was from 3° to 70°. The step size was about 0.033°. The time per step was set at 215 seconds.

The mechanical properties of pristine and NMP-treated rPET fibrous membrane were characterized by an Instron tensile tester (3344, MA USA) with a 10 N load cell. The rPET fibrous membrane specimens were cut into 5 × 47 mm² and tested at room temperature with a displacement rate of 2 mm/min. Both groups of rPET fibrous membrane had three specimens for testing. The Young's modulus of fibrous membranes was calculated based on the obtained stress-strain curves.

2.4. ~~-. Air filtration performance evaluation~~ Filtration Performance Evaluation

2.4.1. ~~-. PM2.5 aerosols filtration~~ Aerosol Filtration

An rPET fibrous membrane was fixed on a 3D-printed 10 cm diameter round holder with a ducted fan driven by a brushless motor in a ~~400-liter~~ 100-l storage box (Argos, ~~UK~~ U.K.) as the confined room (Figure ~~Figures~~ 2a and S1). When the air flow is 3 m/s, the air flux through the membrane and holder is ~~~350~~~ 350 dm³/min because the inner diameter of ducted fan is 5 cm. The pristine and NMP post-treated electrospun rPET fibrous membrane were used for comparing the filtration efficiency of fibers with different surface ~~morphology~~ morphologies. Three groups of both pristine and NMP post-treated were selected with different membrane ~~thickness~~ thicknesses (Table S1 in the Supporting Information) by controlling the spinning time from 1 h to 3 h.



2. ~~-. The schematic~~ Schematic diagrams of air filtration tests: (a) internal air flow for PM2.5 aerosol capturing ~~test~~ test and (b) external air flow for virus interception test.

Moxa is a traditional Chinese medicine ~~which~~ that could be ~~burnt and burned to~~ release a smoke ~~contains~~ containing inhalable particles, carbon monoxide, carbon dioxide, and volatile organic compounds.²¹ Thus, moxa haze could mimic urban aerosol pollutants. A 0.05 g moxa stick (Changzhou, China) was ~~burn for generating~~ burned to generate PM2.5 aerosols. The high-speed ~~airflow~~ air flow motivated by the ducted fan continuously pushes the air to go through the filter ~~membrane~~ membrane so that the aerosol particles in the confined space begin to accumulate on the membrane during the filtration test process as shown in Figure 2a. The downtrend of PM2.5 concentration (~~µg/m~~ µg/m³) was recorded by an air quality monitoring system.

2.4.2. ~~-. Virus spray filtration~~ Spray Filtration

The green fluorescent protein monoclonal antibody (eGFP, ~~Invitrogen, USA~~ Invitrogen) was used to mimic virus due to its availability ~~effor~~ for further observation.²² The eGFP was diluted ~~and generated to~~ generate a mist ~~spray that is sprayed~~ by a household humidifier. The same 3D-printed membrane ~~holder~~ holder and the ducted fan were used to draw the air from the box to the outside. When the air ~~flow~~ flows across the rPET fibrous membrane, the eGFP would be intercepted by the fibers as shown by Figure ~~Figures~~ 2b and S2. The ~~petri~~ Petri dish was placed behind the fan outside the box. The phase contrast light microscopy (Zeiss, Germany) was used to observe the eGFP on the fibrous ~~membrane~~ membrane, and ~~petri-a Petri~~ a Petri dish was used for investigating the filtration performance of pristine and NMP post-treated electrospun PET membranes. The second (electret nonwoven) and third (contact face) layers of commercial surgical face mask (TG, UK EN14683:2019) were used as the control group.

2.5. ~~-. Software~~

Electrospun rPET fiber ~~diameters~~ diameters and mechanical properties were ~~collected~~ determined by applying Image J (version 1.8.0, ~~NIH~~ NIH).

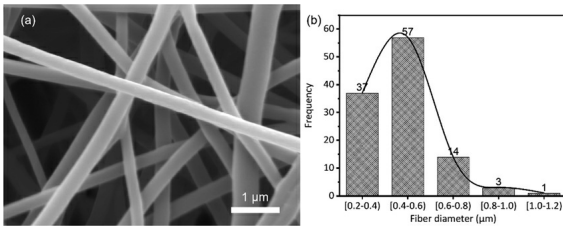
USA). NIH). The results of fiber diameter were presented as $\text{mean} \pm \text{standard deviation}$.

3. Results and Discussion

3.1. Surface Morphology

3.1.1. Surface Morphology of Electrospun rPET Fibers

Figure 3 shows the surface morphology of pristine electrospun PET fibers under $20,000\times$ magnifications. The fibers were randomly oriented. Although most of fibers had smooth surface and round shape, there were few bead fibers observed. It could be attributed to the relatively low polymer concentration. With the fast evaporation of DCM in PET electrospinning solution, the lower solution concentration led to a lower solution viscosity, which was not able to form thin fibers with a higher specific area.

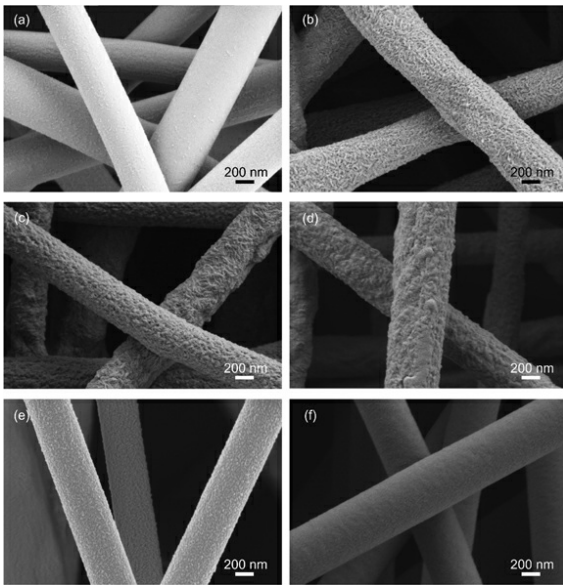


3. (a) SEM images of pristine electrospun rPET fibers and (b) rPET fiber diameter counts.

It could also be observed that the fibers were adhered with each other, which can be attributed to the slow evaporation of TFA due to its relatively high boiling point (72°C) and low vapor pressure (97.5 mmHg, 20°C). The fibers might collapse and were interconnected with each other before TFA was completely evaporated. Furthermore, the relatively lower PET concentration of spinning solution also led to the fibers' adhesion. The lower PET concentration resulted in thinner fiber diameters, which were more likely to collapse and connect with each other. The most of PET fibers (83.9%) were in the size range from $0.2\ \mu\text{m}$ to $0.6\ \mu\text{m}$. The average diameter of electrospinning PET solution at a concentration of 10 wt% was $0.48 \pm 0.15\ \mu\text{m}$. Compared with previous studies, it could be found the different ratios of TFA/DCM solution systems may affect the fiber diameter. Significantly differing with that of TFA (42.1 and $13.4 \times 10^{-3}\ \text{J}\cdot\text{m}^{-2}$), DCM has a lower dielectric constant and a higher surface tension (8.9 and $27.2 \times 10^{-3}\ \text{J}\cdot\text{m}^{-2}$). The higher DCM volume in the electrospinning solution can decrease the stretching of the polymer flow jet during the electrospinning process, then led to the larger electrospun fiber diameters. As a critical scenario of NIPS, the different solubilities and volatilization rates of TFA and DCM make a certain gap between the surface of the fiber and the PET molecular chain interior, which provides the possibility for subsequent solvent-induced recrystallization.

3.1.2. Surface Morphology of Post-Treated PET Fibers

Figure 4 shows the SEM images of electrospun rPET fibers after different solvents treatment under $10,000\times$ magnifications. The immersing duration, 30 min, was same for all specimens treated by different solvents. The recipes for different solvent systems are illustrated in Table 1. The surface of ethanol-treated PET fibers (Figure 4a) became a little rougher than pristine fibers. By contrast, the fibers treated by acetone (Figure 4b) or NMP/ethanol (Figure 4c-f) had a completely different surface morphology. The surface of acetone-treated fibers was much rougher than ethanol-treated and pristine fibers. The rough surface of NMP/ethanol-treated PET fibers had some tiny voids. Therefore, the porous structure on the surface of fibers treated with NMP/ethanol should increase their surface area compared with pristine electrospun PET fibers. However, the pore sizes range from micrometer among adjacent fibers to nanometer inside individual fibers. Therefore, it is difficult to reveal the overall porous structure and surface area by a single method, e.g., pressurized mercury method and N_2 absorption method.



4. SEM images of rPET fibers treated by different solvents for 30 min. (a) ethanol absolute; (b) acetone; (c) NMP/ethanol, (1:1 wt%); (d) NMP/ethanol, (1:1 wt%) after ethanol resin; (e) NMP/ethanol, (3:5 wt%); and (f) NMP/ethanol, (1:3 wt%).

The different parameters of these solvents (Table 2) resulted in the different morphologies of rPET fibers after solvent post-treatment. These post-treatment solvents were carefully selected with the comprehensive consideration of their boiling point, viscosity, volatility, and, most importantly, solubility to the PET molecule. As an improved quantitative strategy for “like dissolves like”, solubility parameter is a widely acknowledged theoretical model for evaluating the specific solubility between polar molecular polymers and solvent.²⁴ Hansen solubility parameters is a criterion that composed of three variables including include molecular dispersion, polar, and hydrogen bonding.²⁵ Hildebrand solubility parameters can be considered as the vector sum of these three variables. The closer the Hildebrand solubility parameters of polymer and solvent are, the more likely they are to dissolve.

2.

Properties of Solvents Used in this study [boiling point; dielectric constant (ϵ); surface tension; viscosity (η); This Study [Boiling Point; Dielectric Constant (ϵ); Surface Tension; Viscosity (η); Hildebrand solubility parameter (δ); vapor pressure] Solubility Parameter (δ); Vapor Pressure]

Solvent	Boiling point (°C)	Dielectric constant (ϵ)	Surface tension (J/m^2)	Viscosity (η , mPa·s)	Hildebrand Solubility Parameter ($MPa^{1/2}$)	Vapor pressure (mmHg, 20 °C)
TFA	72	42.1	0.13	0.93	10.8	97.5
DCM	39.7	9.1	0.27	0.45	4.9	354.8
Ethanol	78.3	24.3	0.02	1.20	13.2	44.6
Acetone	56.1	20.6	0.02	0.31	9.9	184
NMP	203	33	0.41	1.65	11.4	0.3

The solubility parameters between NMP and PET was relatively narrow. NMP is the mainstream post-treatment solvent for PET,²⁶ while acetone is a typical highly polar solvent. The PET fibers would be swollen during immersion in the NMP/ethanol solvents and the NMP solvent-induced re-crystallization happened. solvent-induced recrystallization occurred. Along with the movements of PET molecular chains during forming crystal, crystal formation, solvents were squeezed out of the crystallization part of fibers and phase separation happens. Due to the high boiling point (202 °C), the evaporation rate of NMP was pretty low. The interaction between NMP and PET fibers was strong. It is thus held that the morphology and other properties of fibers after NMP/ethanol treatment might change gradually before totally evaporation of NMP. The specimens treated by NMP/ethanol were rinsed by ethanol with a high evaporation rate for obtaining reliable and consistent results. After ethanol evaporating, porous PET fibers were formed.

By contrast, ethanol has a far different solubility parameter with from PET, which means that it is a bad solvent for the PET molecule.

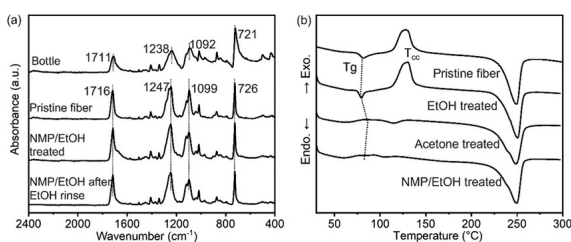
Therefore, the PET ~~re-crystallization~~recrystallization would not happen during ~~immersing~~immersion in ethanol absolute, nor significant morphological changes of rPET fibers was observed.

~~Amorphous~~The amorphous PET film could be swollen and ~~became crystallinity~~become crystalline in ~~the~~ acetone at 25 °C.²⁷ As a result, the ~~morphology~~morphological changes of rPET fibers after post-treatment by acetone might be attributed to the ~~swollenswelling~~ and ~~re-crystallization~~recrystallization during the ~~immersing~~immersion. However, there ~~were~~was only ~~roughness~~a rough surface instead of voids or pores on PET fibers after acetone ~~post-treated~~post-treatment. The possible reason was that the PET experienced the first stage of crystallization during ~~immersing~~immersion in acetone for 30 ~~minutes~~min at 25 °C,²⁸ and no voids appeared during this process.²⁸ ~~Acetone induced re-crystallization~~The acetone-induced recrystallization process could change the morphology of PET fiber ~~membranes~~membranes, and the ~~fibers'~~fibers' surface would ~~turned-into~~become rougher than ~~that of~~ pristine electrospun fibers.²⁹

Figure 4 also compares rPET fibers after post-treatment with ~~different ratio~~NMP/ethanol solvents ~~solvents of different ratios~~. In general, the surface ~~morphology~~morphological changes became invisible with ~~the~~ decreasing amount of NMP in the treatment mixture. Ethanol had limited interaction with PET due to ~~their~~its Hansen solubility parameters. The morphological changes would not happen when ~~fibers~~ immersing ~~fibers~~ in ~~the~~ pure ethanol. As a result, the penetration ability of ~~the~~ solvent mixture was limited when ethanol is the dominant solvent in the mixture. PET fibers would not ~~swell~~swell, and thus ~~re-crystallization~~recrystallization of PET was relatively low, which resulted in ~~a limited~~ a limited ~~morphology~~morphological change of ~~the~~ PET fiber surface. As for temperature, the ~~morphology~~morphological changes after immersing in NMP/ethanol 50:50 mixtures became less significant as ~~the~~ immersing temperature decreases from 25 °C to ~~-20 °C~~-20 °C. Lowering ~~the~~ temperature would decrease the diffusing ability of ~~the~~ solvent molecules. As a result, the interaction between polymer chains and solvent molecules was ~~low~~low, and thus the motion ability of amorphous PET chains was not enough to rearrange and crystallinity was limited, which ~~lead~~led to little surface change.

3.2. ~~Residual solvent~~Solvent of ~~electrospun~~Electrospun rPET ~~fibers~~Fibers

Figure 5a ~~presented~~presents the FT-IR curves for electrospun PET fibers and ~~Coca-cola~~Coca-Cola bottle pieces. For ~~Coca-Cola~~Coca-Cola bottle sample, all characteristic peaks of function groups can be clearly observed, including terephthalic acid ester C=O at 1711 cm^{-1} , asymmetric ~~C-C-O~~C-C-O at 1238 cm^{-1} , stretching ~~O-C-C~~O-C-C at 1092 cm^{-1} , and ~~-CH-CH-~~ at 721 cm^{-1} .³⁰ The peaks of electrospun PET fibers moved ~~towards~~toward a higher wavenumber, but the main peaks of two FT-IR group were similar, which indicates ~~the~~that PET did not decompose during electrospinning.



5. (a) FTIR ~~for Coca-Cola~~spectra of Coca-Cola bottle pieces, electrospun rPET fibers before and after ~~solvents~~solvent treatment. (b) DSC traces ~~for of~~ electrospun rPET fibers before and after treatment ~~of with~~ different solvents for 30 min.

Due to the intensive interaction between PET and NMP, NMP ~~need~~needs to be extracted from the PET fibrous membranes after immersing in the mixture of NMP and ethanol. ~~Regarding~~Because the evaporation rate of NMP at room temperature is relatively low, the specimens ~~were~~ treated by NMP should be examined to determine whether the solvent has been removed successfully. If the membranes after immersing in ~~the~~ NMP/ethanol were dried at room temperature without further processing, NMP would be trapped among the PET fibers due to its low evaporation rate. The ~~swollenswelling~~ and ~~re-crystallization~~recrystallization of PET fibers induced by NMP would happen continuously during this period. It can be seen on the FTIR curve for ~~NMP/ethanol-treated~~NMP/ethanol-treated PET fibers without ethanol ~~rinserinsing~~ that the peak at 1683 cm^{-1} was absent. This peak should be assigned to the C=O bond of ~~-N-C~~the -N-C5O group.³¹ After ~~a~~ twiceringing with ethanol ~~rinsing~~of the NMP/ethanol-treated twice, ~~NMP/ethanol-treated~~ PET fibers disappeared. ~~This~~ indicated that the NMP trapped in the fibers ~~were~~was removed successfully. The changes of the peak at 1683 cm^{-1} may be attributed to two reasons. One is that there was no bond formed between NMP molecules and PET polymer chains during immersion. The other is that NMP was soluble in ethanol, and therefore ~~the~~ ethanol immersion among PET fibers accelerated NMP evaporation.

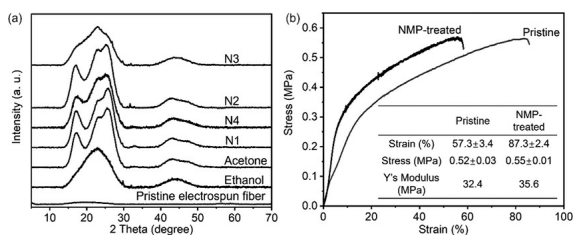
3.3. Thermal-Physical Properties of PET bottles and electrospun rPET fibers

Figure 5b shows DSC traces for electrospun PET fibers before and after treatment with different solvents for 30 min at room temperature. For pristine electrospun PET fibers, there were three major peaks: the glass-transition area (T_g , 81.7 °C), cold crystallization peak (T_{cc} , 128.9 °C), and melting peak (249.7 °C).

The DSC trace for electrospun fibers after ethanol treatment for 30 min was similar to the specimen of pristine PET fibers. This indicated that ethanol treatment for 30 minutes did not change the thermal-physical properties (Table S2) of the electrospun rPET fibers, which means there was no solvent-induced recrystallization during ethanol immersion. This DSC result is consistent with the SEM images, which demonstrated that the ethanol-treated fiber had a similar surface morphology with that of pristine fibers. By contrast, when PET fibers were immersed in acetone or NMP/ethanol solutions, these kinds of solutions can permeate into PET fibers and act as a plasticizer, which can reduce the T_g values of PET fibers, as shown in Figure 5b. The solvent-induced crystallization could be observed from the small area of the crystallization peaks where as indicated by are in the dashed line for NMP ethanol and acetone curves.²⁸ The crystallization of PET fibers during immersion in NMP/ethanol nearly complete showing a near-complete disappearance of cold crystallization peak indicated that some amorphous molecular chains become crystalline. Additionally, the PET fiber after acetone or NMP/ethanol treatment showed a pre-melting peak between 100 °C and 125 °C and a major melting peak at around 250 °C. In their main melting areas, there were two endotherms. The first endotherm reflected the melting behavior of crystallinities of the PET, while the second endotherms reflected the melting behavior of secondary crystallization of the PET during the heating process of DSC.³²

3.4. Relative Crystalline Index of electrospun rPET fibers

The Figure 6a showed the XRD patterns for pristine electrospun PET fibers and the fibers after different solvent treatments. The Coca-Cola bottle specimen had a peak at 25.4° in Figure S1. The pristine electrospun rPET fibers showed a slight halo around 18°. Compared with the XRD patterns of the Coca-Cola bottle, the pristine electrospun fibers improved the amorphous part of raw PET material because the polymer chains consolidated before crystallization due to rapid evaporation of solvents during electrospinning.



6. (a) XRD patterns for pristine and post-treated electrospun PET fibers. (b) Stress-strain curves for pristine and NMP-treated rPET fibrous membrane and their mechanical properties.

The samples E and N3, which were post-treated by ethanol treatment and NMP/ethanol (25/75, w/w), did not have crystalline peaks. They only had two amorphous peaks at around 17° and 22°. This indicated that samples E and N3 had similar structures of the amorphous phase. By contrast, sample N1 treated by NMP/ethanol (50/50, w/w) had crystalline peaks at 19.3° (010), 21.3° (111), and 23.1° (110) and two amorphous peaks. The peak at 23.1° was absent in sample N4, which was treated by NMP/ethanol (50/50, w/w) at a lower temperature (-20 °C). This means that the same solvent system post-treatment at different treated temperatures could not affect the PET crystal structure but changed the amount of crystalline-crystal. The sample N2 that treated by NMP/ethanol (37.5/62.5, w/w) only had a crystalline peak at 22.39°. This might be attributed to the fact that the positions of other two crystalline peaks were unable to resolve due to the low crystallinity.

The acetone-treated sample A had two amorphous peaks at 16.8° and 25.8°. The difference of amorphous peaks between samples treated with acetone and NMP might be caused by the different amorphous structures of samples after

different solvent ~~treatment~~:treatments.

Separation of the amorphous peaks and crystalline peaks in the XRD patterns was ~~used~~performed to calculate the crystallinity degree for PET fibers after solvent post-treatment. The crystallinity degree for fibers after solvent treatments ~~were~~was calculated by the formula (1):

$$CI_{\text{relative crystallinity index}} = \frac{\text{total area of crystalline peaks}}{\text{total area of all peaks}} \times 100\% \quad (1)$$

The protocols to separate the amorphous and crystalline peaks in the XRD patterns of PET fibers were introduced by ~~the work of~~ Murthy *et al.*³³ The model used in ~~the~~ current work was model E, which involves height, ~~position~~position, and full width at ~~half-maximum~~half-maximum (FWHM). The amorphous peaks would be defined from the XRD patterns through applying the amorphous peak parameters from the template, which were used as starting values. ~~Then~~Then, the crystalline peaks would be added in profile analysis of the scans and the parameters of the amorphous peaks would be adjusted to fit the XRD patterns of PET fibers. The two reference amorphous peaks in the PET fibers are located ~~in~~at 16.62° and 20.04°, whose ~~FWHM~~FWHMs correspond to 4.55° and 7.15°. The FWHM for crystalline peak resolved from the XRD ~~need to~~should be below 3°. The background for every sample was subtracted from the XRD scans before profile analysis. The diffraction diagrams with fitted peaks are shown in ~~the~~ Figure 6a. The calculated relative ~~crystallinity~~crystallinities are shown in ~~the~~ Table 3.

3-

Relative ~~crystallinities for Coca-Cola bottles~~Crystallinities of Coca-Cola Bottles and ~~electrospun~~Electrospun rPET ~~fibers~~Fibers before and after ~~different solvent treatments~~Different Solvent Treatments

Sample	Bottle	Pristine fiber	Acetone (A)	Ethanol (E)	NMP/ethanol (w/w)	NMP/ethanol (w/w)		
sample	bottle	pristine fiber	acetone (A)	ethanol (E)	50/50 (N1)	50/50 (N4)	37.5/62.5 (N2)	25/75 (N3)
CI (%)	55.5	0	52.9	0	30.5	25.3	9.7	0

When PET fibrous membranes were immersed in the solvent, the interaction between polymer chains and solvent molecular increased ~~the~~ motion ability of polymer ~~chains~~chains, and ~~made it possible that~~thus polymer chains rearrange below ~~glass transition~~the glass-transition temperature, which ~~result finally results in the formation of crystallization finally~~crystallization. Due to the crystallization, ~~the~~ phase separation happened and formed ~~solvent-rich~~solvent-rich phase and ~~solvent-poor~~solvent-poor phase. After the solvent in the ~~solvent-rich~~solvent-rich phase evaporated, the porous structure of electrospun rPET fibers appeared. Compared with the similar mechanism study on ~~poly(L-lactic acid)~~poly(L-lactic acid),^{9,19} the ~~NMP-induced re-crystallized~~NMP-induced recrystallized PET fibers did not have a significant increase ~~of~~in crystallinity. Combining with ~~the~~ SEM images in Figure 4, it could be assumed ~~the~~that crystallization ~~that~~ induced by NMP cannot penetrate deep into the fiber ~~interior~~interior but only occurs on the ~~fibers'~~fibers' surface. The crystallization of PET during immersion in the solvents could be verified by the disappearance (or decrease) of cold crystallization peak of DSC scanning and change of XRD diagram for electrospun rPET fibers after solvent treatments, compared with pristine electrospun PET fibers. The difference between ~~morphology~~the morphologies of fibers treated with different solvents could be attributed to ~~the~~ difference between ~~the~~ solubility parameter of solvents and that of PET. The relatively small difference in ~~the~~ solubility parameter would induce larger morphological modification.

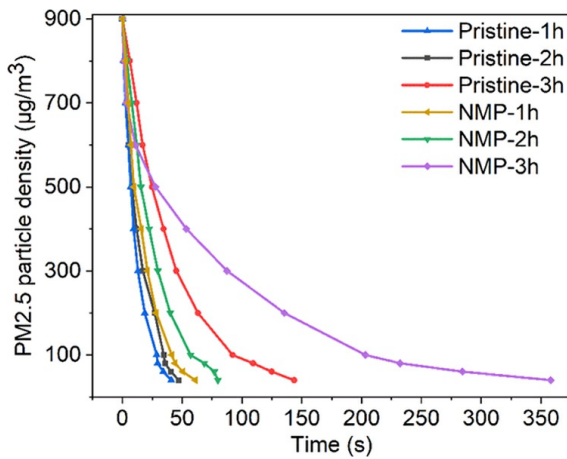
The changes ~~of~~in the mechanical performances between pristine and NMP-treated (N1) samples ~~was~~are presented in Figure 6b. The amorphous pristine electrospun rPET is elastic. ~~Comparing~~Compared with ~~the~~ pristine membrane, the NMP-treated membrane showed higher stress and ~~Young's modulus~~Young's modulus, while its elongation at ~~the breaking point~~break was reduced significantly. This change consists ~~with~~of the increased crystallinity with ~~the solvent-induced re-crystallization~~solvent-induced recrystallization.

3.5- ~~Air filtration performance evaluation~~Filtration Performance Evaluation

3.5.1- ~~PM2.5 aerosols filtration~~Aerosol Filtration

Figure 7 demonstrates the air filtration performances of pristine and NMP post-treated electrospun rPET fibrous membrane samples. For both pristine and NMP post-treated membranes, three groups with different ~~thickness~~thicknesses controlled by setting different electrospinning ~~time~~times were examined. The ~~1 h~~1 h sample was the thinnest ~~one~~one, whereas the 3 h sample was the thickest. Contrary to expectation, the results show that the pristine membranes have better filtration speed than the NMP post-treated ones and the thinner

membranes are better than [the](#) thicker ones.



7. The filtration time for membrane to reduce PM2.5 from 900 to 40 $\mu\text{g}/\text{m}^3$. The membrane thickness increases with [the](#) longer spinning time from 1 h to 3 h.

PM2.5 is the major air pollution concern that industrialized society [need](#) to face. As a typical kind of aerosol pollutant, the threat of PM2.5 to the human body is obvious, especially aerosols have been recognized as the primary transmission route for the spread of COVID-19 during the pandemic from 2020 till now.^{34,35} Despite [of](#) varieties of face masks, fibrous membranes have been also widely applied in other fields [ranging](#) from industry to domestic.³⁶

The performance parameters between PPE masks and [industrial/domestic](#) systems are different. This is mainly due to the [air flow](#) flux of human [breathing](#), and fresh air filtration equipment often differs by more than an order of magnitude.³⁷ As a result, the aerosol filtration mechanisms [that in](#) PPE masks and industrial/domestic systems rely on completely different mechanisms. In particular, the air flow from human breath is [a](#) laminar [flow](#), which could induce the aerosol particles [moves to](#) regularly. The major filtration mechanisms of fibrous membrane including interception and impaction could only happen under the scenario of laminar flow. However, the aerosol [particles'](#) air flow generated by [the](#) brushless duct motor is [pretty](#)-fast. [The flowing eq](#) 2 could be used to identify the situation of air flow at [the](#) duct [motor](#).

$$\text{Re} = \frac{\rho v d}{\eta} \quad 2$$

Where [where](#) ρ is the air density ($1.225 \text{ kg}/\text{m}^3$); v is the air flow [speed](#), which is 3 m/s in this work; and d is [the](#) dynamic viscosity of $1.81 \times 10^{-5} \text{ Pa}\cdot\text{s}$. It could be calculated that the Reynolds number of air flow at [the](#) duct motor is about [10,000](#), which is a typical turbulent flow. But the air flow mechanism in porous materials has not been a consensus yet.³⁸ Referring to the rPET fibrous membrane in this work, the solvent post-treatment process would not only change the [fibers'](#) surface morphology but also make the fibrous membrane more compact. This change [attribute](#) to the lower air permeability or pressure drop of post-treated membranes than that of [the](#) pristine ones. Thus, for the same fibrous membrane samples before and after post-treatment, the sample without treatment has a high air flux and consequently faster filtration efficiency.

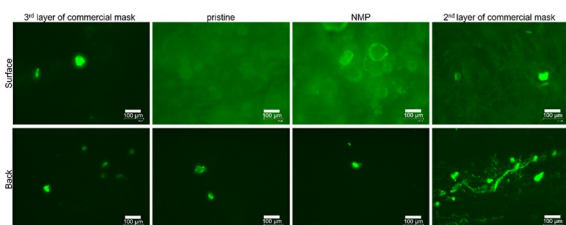
Due to the relatively [fine](#) diameter of PET fibers and the small interior space among fibers, most [of](#) aerosol particles were trapped on the surface of membrane filter (Figure S3). The fast sacked particles would [form](#) a filter cake,³⁹ which could [elevate](#) the filtration efficiency rapidly, especially under [a](#) large flux air flow. Thus, the thicker membrane would be not suitable for face masks due to its higher pressure drop. The video of aerosol filtration process (Supporting [video](#) 01) recorded the fast [accumulated](#) on the surface of rPET fibrous [membrane](#), which could prove this mechanism. A [longer-time](#) filtration test demonstrated the stability of this membrane in the same video.⁴⁰

3.5.2. Fluorescent [protein-mimicking-virus-filtration](#) Protein Mimicking Virus Filtration

Viruses, including SARS-COV-2 coronavirus, which has caused a pandemic in the past one and a half years, are essentially [sub-micron](#) protein macromolecules.⁴¹ Extensive studies have confirmed that human body fluids [the](#) containing SARS-COV-2 virus are the main way to spread COVID-19.⁴² Moreover, this fact has been recognized and taken into policies by [the](#) governments all over the

world.^{43,44} Considering the risk of virus handling, this experiment uses the fluorescent protein mimic virus commonly used in biological laboratories to test the performance of the virus-containing water mist filtration of the rPET fibrous membrane.^{22,45} This study uses an atomization device to disperse the fluorescent protein solution of appropriate concentration into small droplets, which simulates the human body fluids such as those from coughing or sneezing to spread the virus (Supporting video Video 02). As shown in Figure 2b, the experiment used an external air flow to guide the water mist-containing fluorescent protein to pass through the fibrous membrane filter. After filtration tests, a fluorescence microscope was used to observe the residual fluorescent protein on the surface of membrane filters and the receiving plates (cultivation dishes) installed behind the fan. If most of the protein particles remain on the surface of the fiber membrane and there is almost no fluorescent protein on the petri dish, it means the surface of the testing sample retains most of the contaminants and has good filtering performance, and vice versa.

Figure 8 presents the results of the above experiment. The conventional three-layer non-woven surgical mask was involved in the experiment as a comparison group. Overall, rPET fibrous membrane, whether it is pristine or NMP-treated, has achieved good results comparable to the second layer of the mask. The second layer is the vital layer that applies the electret technology in surgical masks to intercept pollutants. On the surface of the second layer of PET membrane samples and surgical masks, it was obviously observed that a large number of fluorescent protein particles are attached to the fiber surface. The third layer of surgical masks is mainly a layer that plays a role of moisture absorption in commercial design.³⁷ The third layer of surgical mask has almost no interception effect of fluorescent protein. By observing the surface of the petri dish behind the ducted fan, we could conclude that the third layer of the surgical mask cannot filter most protein particles. Although the second layer of the surgical mask has a good filtering efficiency, some fibers were detached from the surface of the membrane due to continuous airflow, while some fluorescent proteins are carried along with these fibers. This result proves that the melt-blown electret filter membrane cannot be used as a safe virus protection device alone but needs the assistance of the back layer of non-woven fabric. On the contrary, the rPET fibrous membranes fabricated by electrospinning are more stable in the working environment. These electrospun membranes not only can effectively intercept the virus, but also the membrane itself was not damaged. Further comparison shows that fewer fluorescent proteins can be passed through the NMP-treated sample. Therefore, the filtering capability of post-treated high-roughness fibrous membrane is better. This may be attributed to its rougher fiber surface, which gives the membrane more opportunities for interception.



8. The microscope images of fluorescent proteins mimicking virus which were captured on the surface of membrane filter and left on receiving petri dish.

4. Conclusions

In brief, the rPET fibers and fibrous membranes were successfully electrospun by applying TFA/DCM (50/50, w/w) in this work. Observed by SEM images, the hypothesis of controlling PET fibers the surface morphology of PET fibers by adjusting post-treatment parameters was proved. It can be found that either NMP or acetone under appropriate conditions can generate obvious porous structure on the surface of rPET fibers. It was confirmed by further characterization that the morphological change of electrospun rPET fibers was attributed to solvent-induced crystallization mechanism. The pristine PET membranes could filter PM2.5 aerosol particles, but the NMP post-treated membrane shows the better filtration capability for protein capturing. Therefore, the electrospun rPET fibrous membrane developed in this work can be applied for the effective filtration of various pollutants including PM2.5 and viruses and is especially suitable for potential applications in industrial or domestic high-flux filtration equipment.

Supporting information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsami.xxxxxx>.

~~The thickness~~[Thickness](#) of rPET fibrous membranes for aerosol filtration; ~~Thermo~~[thermal](#) properties of PET bottle, pristine and post-treated rPET fibrous membranes; ~~The~~ optical photo of internal air flow for PM2.5 aerosol capturing and virus interception air filtration test; ~~and~~ SEM image of rPET fibrous membrane after aerosol filtration ~~test; and The videos of PM2.5 aerosol capturing and virus interception air filtration experiments. test~~ (PDF)

[Videos of PM2.5 aerosol capturing and virus interception air filtration experiments](#) (MP4)

(MP4)

The ~~3D printed~~[3D-printed](#) model (Shapr3D) used in this ~~manuscript~~[study](#) is freely available at: <https://github.com/Airslashcn/3D-printing-for-aerosol-filtration>.

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