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A Novel Preparation of Anti-Layered Poly (vinyl alcohol)-Polyacrylonitrile (PVA/PAN) Membrane for Air filtration by Electrospinning

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The novel 3D filtration media for airborne particles has been prepared via electrospun technique, the PVA and PAN were used as reinforcing agent and bulk material for filtration membrane respectively. In order to improve the filtration efficiency and mechnical property of membrane, the membrane was modified by the certain concentration mixture of GA, acetone and acetic acid, which could reduce the amount of hydroxyl groups, and then promote its stability and increase the compactness via bond between fibers. Simultaneously, the mechanical property for the membrane had been measured by tensile tester and the results demnostrated that the maximum strain and toughness reached 144.58% and 5.33 jg^{-1}. After that, the membrane was used as air purfication material for testing of air particles content and the particles intercept principle has been discussed. The filtration performance of membrane demonstrated the membrane possessed excellent removing efficiency for the air suspension with lower pressure drop compared with commercial masks under same testing conditions. And the filtration efficiency for the particles sized 0.3 to 7 μm were about 99%. Furthermore, we also developed an effiective avenue to fabricate the filtration medium based on the steel wire for applications in ultra condition.

Introduction

Air purification has attracted more attention following the increasingly serious environment problems.[1] The particulate matter (PM) pollution had been confirmed as major cause of jeopardizing the human respiratory tract, which was one of the acute challenges facing the world today. The industrial production and automotive emissions have aggravated the phenomenon of excessive fine particles.[2,3] As we all know that PM are mixtures of small particles absorbed by lots of viruses and bacteria, therefore, it poses a serious threat to public body health and it is necessary to avoid these polluted materials entering into our body organs. Recently, the hazy days become more frequent than before, the visibility decreased severely because of the fine particles (The aerodynamic diameter ≤2.5 μm), especially for the northeast of China in winter, the particulate matter quantity in the air would increase greatly for warmth via burning fossil fuel and vehicle emission, which caused great impact on the climate and ecosystems of environment.[4] In order to protect the health of people, lots of products about purifying air have been produced. However, the filters based on the conventional fibres (e.g., glass fibres, melt-blown fibres, and spun-bonded fibres) will not satisfy the demand of people for air purification, especially for removing the fine particles with diameters in the range of 300–500 nm suffused in the atmosphere.[5-8] Moreover, the bulkiness, thickness of the conventional filter medium would be the deficiency for the applications under specific conditions, such as tiny filter devices. Therefore, the characteristic of thinner fibres may possess the great potential for maximizing the filtration efficiency as much as possible. To date, enormous efforts have been devoted toward fabricating the high filtration efficiency medium, and it is noting that the sub-microfiber filter media have been demonstrated as excellent separation material for the remarkable specific surface area, high porosity and interconnected porous, especially for the controllable fibres diameter.[9] As for the preparation of sub-micro fibres, the electrospinning technique would be the outstanding candidate to create fibrous membranes based on several fascinating features, such as remarkable specific surface area, high open porosity, low basis weight and interconnected porous structures.[10,12] Up to now, there were lots of polymers which had been employed as spinning materials for removing the particles in the air purification.[5, 13] However, these membranes still have some defects during the process of application, such as poor mechanical property, which have greatly hindered its applications in the field of filters. Additionally, the membranes tend to be layered when it gets to a certain thickness.[14] Herein, we firstly presented a novel and cost-effective strategy to fabricate the composite membranes through double jet-electrospinning, which showed high efficiency filtration for air particles. As we all know that polyvinyl alcohol (PVA) and polyacrylonitrile (PAN) are applied widely in generating the organic fibres by electrospinning, which have their own advantages in the applications, respectively.[15,16] As for the membrane, the PAN fibre serves as the bulk material for filter owing to the chemical stability and outstanding weatherability in the applications.[17] In order to improve the mechanical property of the membrane, enormous studies have attempted to resorting to the blending polymers fibres during the electrospinning process and thermal treatments for the composite membrane.[18,19] For an

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example, Ding et al. fabricated FPUPAN/PVB membrane crosslinked by thermally treatment, which has greatly enhanced the mechanical properties.[20] Meanwhile, we have prepared the PAN/PVA membrane crosslinked by chemical bond, which could avoid layering and osteoporosis of surface comparing with. During the process of fabrication, PVA was used as reinforced matrix to immobilize the PAN fibres through connection points after crosslinking; the schematic diagram of experimental set-up is shown in Fig. 1.

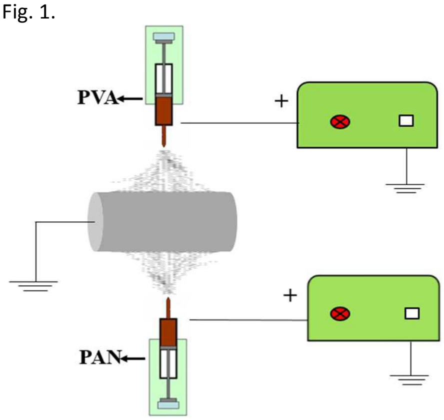

Fig. 1 The schematic diagram of electrospinning set-up

Then, the composite membranes were immersed in the certain concentration of solution mixed with acetic acid, glutaraldehyde (GA) and acetone.[21] The glutaraldehyde would capture the hydrophilic functional groups of PVA and form the bonding structures among the PVA fibers. (Fig. 2) Because of the interpenetrating PVA and PAN fibers, these crosslinking parts would assemble into stable filtration medium, which possess the features of high porosity and packing density via optimizing the contents ratio of PVA and PAN fibers. As we can see from the ESI (Fig. S1), the crosslinked composite membrane was kept in good morphology and crossed each other via connect points of PVA fibres comparing with uncrossliked membrane. This is because the hydroxyl groups of PVA fibre would react with the molecules of GA and form the bond to link the two parts. The detailed plots of crosslinking were shown in the Fig. 2; there are two aldehyde groups at end of GA, which would react with hydroxyl of PVA and produce bridge pattern after the process of acetal reaction. The fibers of PVA would be intertwined each other, and improve the strength and thermal stabilities in some certain degree.[22]

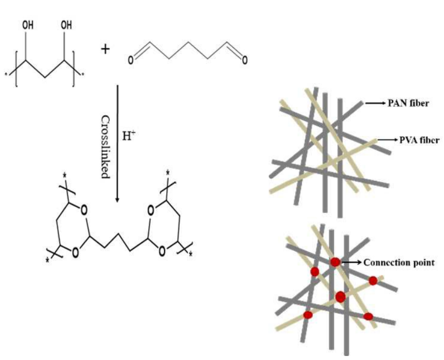

Fig. 2 The mechanism of crosslinking and fibres fixed model

In this contribution, we presented the fabrication of multilevel structure filtration medium via multi-jet electrospinning process. Moreover, we also resolved the delamination of single fibrous membrane and contact problem of film-upholder interface in the facile way. Compared with the technique of thermal treatment for the membranes, this approach of preparing the composite membranes has great potential in the future. Because the materials could keep the morphology and chemical properties of the composite membranes.

Experimental section

Materials

Polyacrylonirile (PAN, Mw = 90 000,) and Polyvinyl Alcohol 1799(PVA, Alcoholysis: 99.8–100%) were used in this experiment, which were purchased from Aladdin. Glutaraldehyde (GA, 50%) and acetic acid (99.7%) were purchased from Beijing Chemical Works. The acetone and N, N-dimethylformaide (DMF) were obtained from Sinopharm chemical reagent Co. Ltd. Stainless Steel Mesh (SSM, 300 mesh) was bought from the commercial market. Ultrapure water was used in all experiments. All chemicals were used without further purification.

Methods

At firstly, the PAN was dissolved in the DMF with the mass percentage concentration of 14 % and the PVA was dissolved in the deionized water with the concentration of 12%. Then, the two different solutions were transferred into two different syringes to generate polymer fibres by electrospinning equipment with boost devices. During the electrospinning process, the flow rates of PAN and PVA were controlled at 1.0 mLh^{-1} and 0.2 mLh^{-1} respectively by peristaltic pump. The high voltage applied to the needle of the PAN solution syringe was 15 kV, and distance from the needle tip to rotating roller covered by SSM was 20 cm. Simultaneously, the high voltage applied on the PVA syringe was 10 kV and the distance was 20 cm. After that, the composite membranes were crosslinked by the mixture of GA, acetic acid and acetone for 1 hour. Moreover, the SSM was immersed in the absolute ethyl alcohol to wash contaminants adhering on the surface for 2h. Then, the SSM would be put into dilute PVA solutions with the concentration of 2% for 2 hours. After that, it was used as collector for the composite fibres and transferred into the gas mixture of GA and acetic acid.

Characterizations and measurements

The membranes were observed by Scanning Electron Microscope (SEM, SHIMADZUX-550) after applying a gold coating. The Fourier Transform Infrared spectra of the membranes were obtained from the Paragon 1000 Spectrometer (Perkin Elmer) at a signal resolution of 1 cm^{-1} within the range of 400–4000 cm^{-1}; the thickness of samples was measured by micro-screw meter (0–25, Shanghai). We used the tensile tester to measure the mechanical property of the membranes with stretching velocity of 5 mm min^{-1}. The contact angle of membrane interface with drop of water was measured by Contact Angle Analyzer (Kino SL200B, USA). In order to the authenticity of the test results, the contact angles were measured for three times and the average values were recorded. Moreover, the filtration performance of the membrane was tested in the real polluted air environment, the $PM_{2.5}$ diffused in the air on that day was at $PM_{2.5}$ index > 200. The membrane was used as filters to measure the Air particle index for one day. And ultimately, to evaluate the filtration performance of the membranes, the air particle counter (MET ONE 237B) was used for detecting the number of particles through the samples and the removal efficiencies are calculated by comparing the PM particle number concentration without air filters. The pressure drop can be calculated from the height difference marked in the tube U, the schematic of detection process was shown in the following schematic:

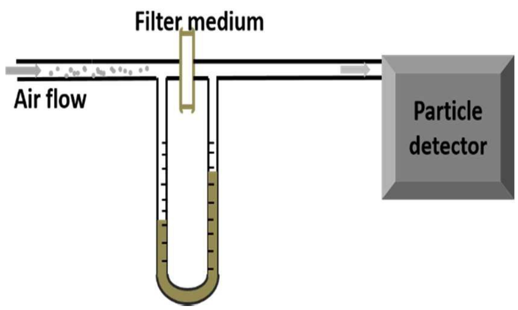

Fig. 3 The schematic diagram of detection device for airborne particulate matter

Results and discussion

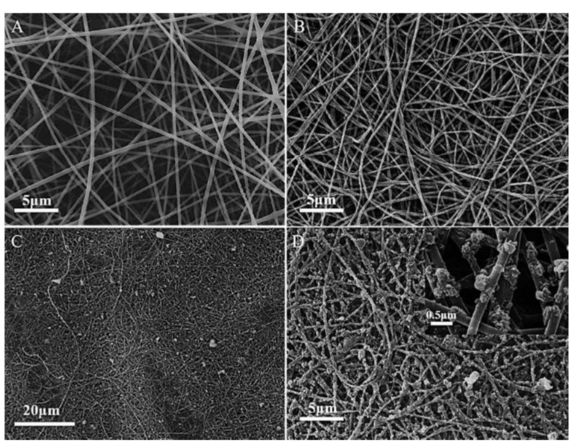

Fig. 4 The morphology of membranes (A The PAN/PVA membrane; B The morphology of crosslinked PAN/PVA membrane and C The low magnification morphology of composite membranes used for 24 hours' filtration in the air; D The SEM image for the back side of membrane used for 24 h. The insets were the corresponding pictures for the membranes respectively)

As shown in Fig. 4, the morphology of membrane was observed from SEM images. By adjusting the concentrations of polymers and parameters of electrospinning, the average fibres' diameter could be precisely controlled. The diameter of fibre is critical factor for the filtration efficiency in the application.[23] As for the PVA fibres, it exhibited smooth, uniform morphology and narrow diameter distribution from 0.55~0.65 μm. Comparing with PAN fibers, the PVA fibers play a great role in the enhanced tensile strength. The diameter of PAN fibres ranged from 0.25~0.35 μm, which was used as the primary materials for removing particles and about 80% in the mass ratio compared with PVA. The Fig. 4B is the SEM image of membrane after crosslinking. As observed from the morphology of cross-linked membrane, the packing density of film has been increased obviously and the aperture decrease greatly for the formation of connect points between PVA fibres via crosslinking. The high magnification of SEM image for the cross-linked membrane presents in the Fig. S1, the bonding structures have been formed among the adjacent fibres of PVA, which was highlighted by the red dotted circles. Compared with uncross-linked fibres, the PVA and PAN fibres' morphology remained almost unchanged. However, there are some changes for the structures between the fibres. For an example, the ribbon-shaped bonding structures formed between the PVA fibres as shown in the yellow dotted rectangle (Fig. S1). On the basis of the real weather situation, the membrane was tested for filtration performance under the condition of hazardous level equivalent to the $PM_{2.5}$ index >200 for 24 hours. Fig. 4C is the low magnification of SEM image of membrane used for 24 hours. The particulate matters in the air were captured by the fibres. The high magnification of SEM images were shown in the Fig. 4D. From the morphology of intercepted particles, the general capture mechanism for airborne particles is that the particles would aggregate together and wrap around the fibres tightly, which indicates that the roughness of fibre may possesses more excellent filtration efficiency for the airborne particles than that of uniform fibres. The Fig. S2 proofed the suspicion and the image reveal that the particles captured were centralized at the parts of one fibre. As we all know that the principles and fundamentals of airborne filtration have been validated with respect to micron particles, such as interception effect, inertial deposition and gravity effect. However, the mechanisms associated with the application of filters for airborne nanoparticles are still uncertain, thereby, the experiment may provide a further research model in the future.[24,25]

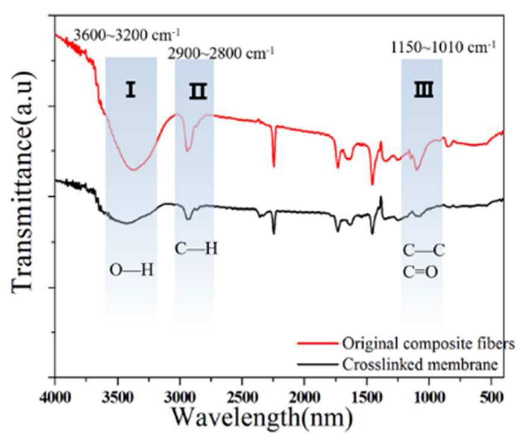

Fig. 5 The contrast FT-IR patterns of membranes (red curve for uncrosslinked membrane and the black for crosslinked membrane)

Table 1 The tensile testing of electrospinning membranes

Samples code	Thickness (mm)	Density (g cm^{-3})	Stress (M Pa)	Strain break (%)	Area (M Pa)	Toughness (J g^{-1})
①	0.038±0.010	1.18±0.07	5.01±0.14	134.76±7.2	4.26±0.91	3.61±0.27
②	0.047±0.003	1.21±0.01	6.93±0.57	182.51±11.3	7.55±0.37	6.23±0.52
③	0.043±0.001	1.23±0.04	8.19±0.31	144.58±4.3	6.56±0.16	5.33±0.14

In order to measure the changes of membrane after crosslinking, Fourier Transform Infrared Spectroscopy (FT-IR) was used to characterize the presence of specific chemical groups in the membrane. The red curve presents the FT-IR pattern of membrane before crosslinking, and the black curve is the FTIR spectra of crosslinked membrane, which shows some changes in the specific peaks compared with original membrane. As shown in the Fig. 5, the band observed from 3600-3200 cm^{-1} represents the peak of stretching O–H group from the intermolecular and intramolecular hydrogen bonds, which has a considerable reduction in the intensity of O–H because of reaction of PVA and GA. The result indicates that a possible formation of acetal bridges as we can see from the mechanism of crosslinking (Fig. 2). The region Ⅱ refers to the stretching C–H from alkyl groups, and there is an unconspicuous peak about C–H for aldehyde in the cross-linked membrane, which is evidenced the crosslinking reaction. The range from 1150 cm^{-1} to 1010 cm^{-1} are related to the symmetric C–C stretching mode or stretching of the C=O of a portion of the chain according to the literature.[22, 26]

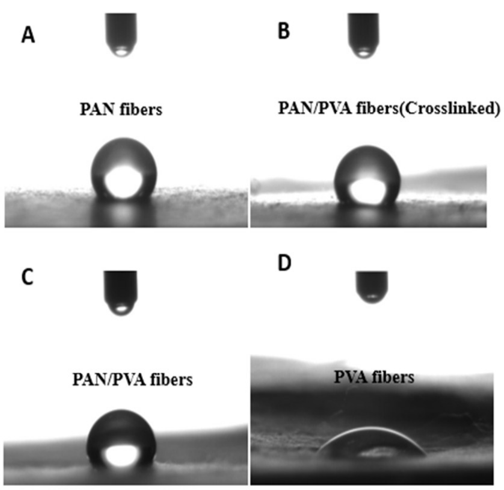

Fig. 6 contact angle of electrospun membrane (The insets of marker were corresponding to the samples and the photos of water drops on the part of flat films

Immediately following, the wettability of the membranes surface was measured for the importance to the filtration materials.[27] In order to investigate the wetting behavior of the electrospun membranes and crosslinked membranes, the contact angle hysteresis values were measured. As shown in the Fig. 6, the wettability of the membranes surface should be hydrophobic except for PVA film. Fig. 6 show the wetting property of the discussed membrane flat surfaces. As observed from the Fig. 6A, the contact angle for a water droplet on the PAN film surface was 122.43±1.3°, indicating that the membrane surface was hydrophobic comparing to the PVA film surface[28] (contact angle: 42.32±2.2 in Fig. 6D). Fig. 6B and 6C reveal that the contact angles of composite films were 117.84±3.2° and 110±4.6°, respectively. The results have good agreement with the FT-IR, because the hydrophilic groups (-OH) of PVA have decreased after crosslinking by the mixture of acetic acid and GA. The hydrophobic property could keep the filtration materials from attaching by the liquid drop, which plays a great role in the applications.[29]

Ordinarily, the mechanical property of cross-linked membrane is another important factor to evaluate the property of materials in the process of applications, therefore, tensile testing was conducted on the as-prepared samples.[30] The results are shown in the Fig. 7 and Table 1. Compared with the tensile stress–strain curves of various membranes, we can clearly observe that crosslinked membrane corresponds to the highest tensile strength (8.19 MPa) among three samples. With regard to the phenomenon, the PVA blended in the membrane played a significant role in the enhanced mechanical properties. Based on our previous research, the polymer network of PVA could be tailored into three dimensional network structure in the molecular level, which would enhance the strength a lot for the whole membrane.[18] Because of the interpenetrations of PAN and PVA fibers, the PVA would immobilize the PAN in some degree by forming the connection points. Furthermore, owning to the adding of PVA, the toughness of PAN membrane also has been improved obviously from 3.61 J g^{-1} to 6.23 J g^{-1}, the detailed data were listed in the Table 1.

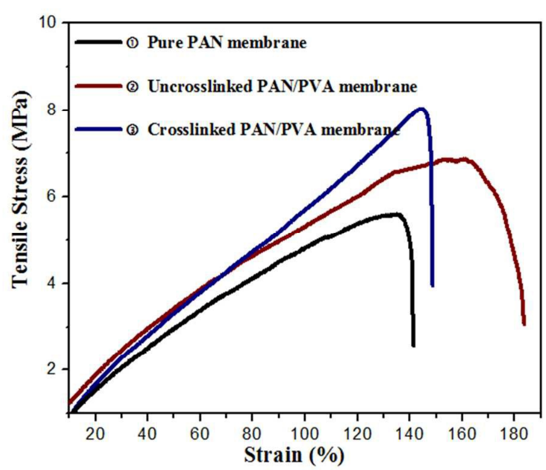

Fig. 7: the curves of tensile tests for different membranes

Table 2 The detailed plots of membrane for filtration of airborne particles

Particle size (µm)	0.3	0.5	0.7	1	2	5	Thickness(mm)	△P (Pa)
Commercial-1(DMM)	0.8389	6.4943	14.7786	24.9603	43.1035	70.8861	0.336±0.002	536±7
Commercial-2(3MM)	61.3765	88.7457	94.7796	95.8468	98.7862	99.9945	1.267±0.005	878±3
ESM	99.9961	99.9971	99.9985	99.999	99.9994	100	0.020±0.001	418±23

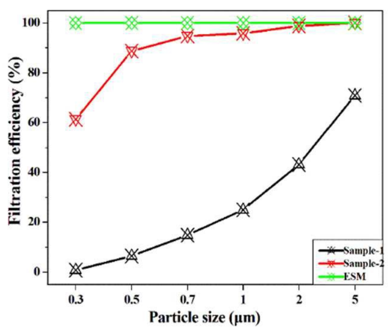

Fig. 8 The comparison of filtration efficiency of three samples

As the concentrations of airborne particles increased with the development of industrialization, the concerns had been arisen with respect to the negative impact on human health, because these particles of air could cause adverse health problems directly owing to the particles acting as carriers of toxic elements.[31,32] It is obvious that we should emphasize the importance of controlling the particle number concentration out of our body. In order to investigate the filtration efficiency of the samples, the air particle counter (MET ONE 237B) was used for detecting the number of particles through the samples. Comparing with the filtration efficiency of two commercial masks, the electrospinning membrane (ESM) exhibited the high efficiency for removing airborne particles. The detailed parameters about the test were listed in ESI (Table S1). The performance efficiency for removing particles was shown in Fig. 6, the removal efficiency of ESM for particles ($PM_{\leqslant 1.0}$) had reached to 99.99%, which was higher than that of the other two commercial masks. Furthermore, the average pressure drop of ESM was just about 418 Pa with the air flow velocity of 0.42 m s^{-1}. For comparison, the filtration efficiency of DMM and 3MM were just about 0.8389% and 61.3765% respectively for the size of particles ⩽ 0.3μm, and the detailed results were listed in the Table 2. Hence, the composite membranes possess great potential as filter media.

In general, the mechanical properties of filtration have some weakness for the demand of filtration applications under extreme conditions, such as high air flow and working pressure etc. As for the previous researches, the enhanced mechanical properties were modified by introducing blend polymers, thermal treatment. However, to our best knowledge, there are few efforts have been devoted to fabricating the 3D electrospinning membranes based on the stainless steel mesh for the hardship of close-knit between the interfaces of membrane and SSM. As shown in the Fig. S3, the modified SSM is the collector to receive the blending fibres of PVA and PAN with the thin layer of membrane. And the light part marked by red dot circle is the joint of stainless steel wires. So as to realize the close integration of interfaces of membrane with SSM, the SSM was rinsed with absolute ethyl alcohol and then modified by dilute PVA solution. After that, the treated SSM had been used as collector for the fibres of PVA and PAN. Afterwards, the filtration medium was cross-linked by the atmosphere of GA and acetic acid. As we can see from the pictures of interfaces of membrane with SSM compared with uncross-linked and cross-linked, the cross-linked composite membrane adhered on the surface of SSM tightly, which would stop the membrane from falling off from the SSM. This is because the PVA fibre would react with the PVA adhering on the surface of SSM immersed in the dilute PVA solution, which would lead to the cohesion of membrane with SSM.

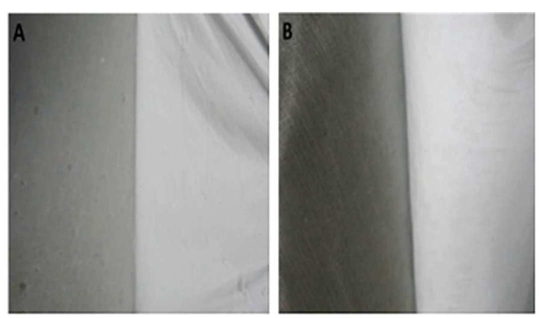

Fig. 9 The contrast of avulsion interfaces for membranes and SSM (A and B were cross-linked membrane and uncross-linked based on the SSM, respectively)

Conclusions

In summary, the novel filtration medium has been fabricated via electrospinning technique, which exhibited significant filtration efficiency comparing with commercial products. The mechanical and wettability of membrane have been improved by physically structures of blend polymer fibres and chemically cross-linked networks between the intertwined PVA fibres. Additionally, we used stainless steel mesh modified by diluted PVA solution as substrate for supporting the membrane, which is beneficial for the applications for filtration material in the extreme conditions. PVA fibres have great influence on the qualities of membrane, especially for the mechanical property, which serve as reinforcing agent after crosslinking by the liquid and atmosphere of GA and acetic acid for the membrane. Overall, the method of preparation multi-layers of membranes provides versatile strategy for further design and develop functional composite fibrous membranes for more mainstream applications in filtration technology, which could take advantages of all the features of polymer fibres to ensure the integrated properties of materials.

Acknowledgements

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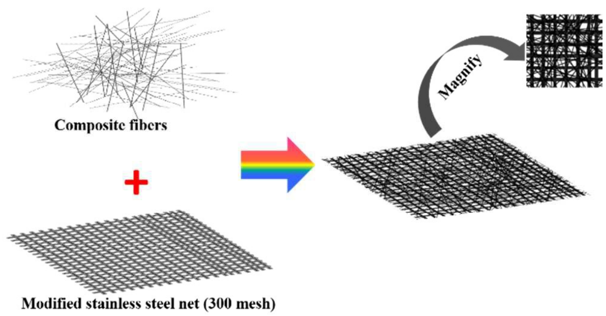
Graphical Abstract



The schematic of preparation for PAN/PVA membrane deposited on the stainless steel mesh modified by dilute PVA solution