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Improving Tensile Properties of Flexible Conductive Polyacrylonitrile@Carbon Nanotube Nanofiber Membrane by Cellulose Nanocrystal

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Abstract: The flexible conductive nanofiber membrane is widely used in the field of wearable electronics. High tensile properties of electrospun nanofiber membranes are essential for their successful commercial application. With cellulose nanocrystal (CNC) as the reinforcement, the flexible conductive polyacrylonitrile (PAN)/CNC@carbon nanotube (CNT) nanofiber membrane is electrospun from the PAN solution containing suspended CNC and impregnated with the CNT solution. The structure and properties of nanofiber membranes are studied. The results show that with the increase of the PAN mass fraction, the viscosity of the electrospinning solution increases, leading to an increase in the nanofiber diameter. When the mass fraction of PAN is 12%, PAN/CNC nanofiber membranes at different CNC mass fractions are successfully prepared. The structure and properties of PAN/CNC nanofiber membranes are affected by the addition of CNC. As the CNC mass fraction increases, the nanofibers become thicker, the nanofiber diameter distribution widens, and the tensile strength first increases and then decreases. When the mass ratio of PAN to CNC is 4∶1, the tensile strength of the PAN/CNC nanofiber membrane is the highest, and it is higher than that of the PAN nanofiber membrane. After impregnating the PAN/CNC nanofiber membrane with CNTs, the tensile strength of the nanofiber membrane increases to 3.12 MPa and the surface resistivity is 64 Ω/cm^2. The flexible conductive nanofiber membranes would be used in energy storage and sensing fields, and the study might provide a strong base for their future development.

Keywords: cellulose nanocrystal (CNC); flexible conductive nanofiber membrane; tensile property; carbon nanotube (CNT); electrospinning; polyacrylonitrile (PAN)
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0 Introduction

Flexible and stretchable wearable electronics have significant application potential in various fields, such as bioelectronics[1-2], human-computer interaction[3-4], smart living[5-6], electronic skin[7-8] and healthcare[9]. However, conventional microelectronics manufacturing processes brittle semiconductor materials and is not suitable for the fabrication of flexible and stretchable wearable electronics[10]. Electrospinning, a simple, universal and low-cost technology, emerges as a versatile and cost-effective solution and is used for preparing continuous nanofibers[11]. Electrospun flexible nanofiber membranes have been extensively used in biomedicine, filtration and protection, energy storage and energy catalyst[12-14]. Li et al.[15] prepared the biocompatible poly (lactic acid)/wool keratin/carbon nanotube (CNT)@CNT fiber membrane via electrospinning and immersion for a pressure electronic sensor with flexibility and conductivity. CNTs have excellent properties, such as high conductivities and high elastic moduli. Zhang et al.[13] prepared the polyacrylonitrile (PAN)/$LiFePO_4$ precursor/CNT membrane via electrospinning, and the membrane was used as the cathode of the lithium-ion battery after heat treatment. PAN membranes have gained significant attention due to their exceptional properties. They are synthesized via fabrication techniques such as phase inversion and electrospinning, and possess excellent mechanical properties, thermal stability and chemical resistance. PAN membranes have been extensively studied for various applications like oil-water separation, heavy metal ion removal, water purification, hemodialysis and lithium-ion batteries[16].

Cellulose nanocrystal (CNC) is extracted from plant fibers and has received a tremendous amount of interest in recent years due to its unique properties and potential applications[17-18]. CNC is a class of fascinating bio-based nanoscale materials, and possesses high strengths, rigidity, high moduli, adaptable surface chemistry, low

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densities, bio-compatibility and renewability, making it an attractive nanomaterial for use in various fields such as renewable energy, electronics, biomedical engineering and pharmaceuticals[19-24]. Specifically, CNC is a needle-shaped or rod-like nanoparticle, and exhibits a high aspect ratio and high tensile strength. Surface hydroxyl groups on CNC could provide active sites for hydrogen bonding with the hydroxyl groups of hydrophilic polymer matrices and physical interlocking with the non-polar matrices, to induce the reinforcing effect in the polymer. The large interfacial area and strong interactions between the reinforcement and the polymer matrix would enhance nano-confinement effects that substantially improve mechanical properties[25-27]. CNC has been successfully used to reinforce various electrospun polymer nanofibers. Dong et al.[28] produced uniform fibers composed of poly(methyl methacrylate) (PMMA) and CNC by electrospinning. Xiang et al.[29] improved the tensile strength of the electrospun nanofiber membrane by the incorporation of CNC into poly(lactic acid). Besides, the abundant hydroxyl groups on the CNC surface enable the material to possess high reactivity, contributing to the possibility of extensive chemical modification[27].

In this study, based on the principle of electrospinning, CNC reinforced PAN (PAN/CNC) nanofiber membranes are prepared. After CNT impregnation, the flexible conductive PAN/CNC@CNT nanofiber membranes are prepared. The effects of CNC addition and CNT impregnation on the structure and properties of electrospun nanofiber membranes are studied.

1 Materials and Methods

1.1 Materials

PAN (the weight-average molecular mass is 150 000 g/mol) was provided by Sigma Aldrich, USA. *N*, *N*-Dimethylformamide (DMF) was provided by Shanghai Richjoint Chemical Reagents Co., Ltd., China. CNC was provided by Shansi Technology Co., Ltd., China. CNTs were provided by Nanjing XFNANO Materials Technology Co., Ltd., China. Sodium dodecylbenzene sulfonate (SDBS) was provided by Beijing Tong Guang Fine Chemicals Company, China.

1.2 Sample preparation

1.2.1 Preparation of electrospinning solution

PAN electrospinning solutions at PAN mass fractions of 10%, 12% and 14% in DMF were prepared at 60 ℃ with magnetic stirring by an S10-3 stirrer (Shanghai Sile Instrument Co., Ltd., China) for 24 h.

PAN/CNC electrospinning solutions at PAN/CNC mass ratios of 2∶1, 3∶1, 4∶1 and 5∶1 were prepared as follows by adjusting CNC mass fractions. CNC solutions in DMF were prepared by a KQ-500DE ultrasonic device (Kunshan Ultrasonic Instrument Co., Ltd., China) for 2 h, and then stirred by the S10-3 stirrer for 2 h. The PAN solution (a mass fraction of 12%) was mixed with the CNC solution by the VORTEX3 vortex mixer (IKA, Germany).

1.2.2 Preparation of electrospun nanofiber membranes

PAN and PAN/CNC nanofiber membranes were prepared by using high-voltage electrospinning. The electrospinning solution was loaded into a 10 mL syringe with a capillary tip (a diameter of 0.51 mm). The flow rate of the solution was 1 mL/h. A variable high-voltage power supply (EST804A, Tianjin Dongwen, China) was used to provide an electric potential of around 15 kV. The tip-to-collector distance was 15 cm. The nanofibers were collected on a flat and nonstick release paper. The nanofiber membrane was removed from the release paper and then dried in a DHG-9035A oven (Shanghai Yiheng Technology Instrument Co., Ltd., China) to remove the residual solvent.

1.2.3 Preparation of flexible conductive PAN/CNC@CNT nanofiber membranes

The electrospun PAN/CNC nanofiber membrane was impregnated in the CNT solution to obtain the flexible conductive PAN/CNC@CNT nanofiber membrane.

1) Preparation of the CNT solution. The electrospun nanofiber membrane of a certain size was prepared and weighed. Deionized water was weighed according to a certain bath ratio(1∶100 and 1∶50). The mixed solution of SDBS (100 mg/mL) and CNT (2 mg/mL) was prepared with the KQ-500DE ultrasonic device for 1 h and then mixed on the VORTEX3 vortex mixer to form a homogeneous solution.

2) Functionalization of the electrospun nanofiber membrane. The prepared electrospun nanofiber membrane was fixed on the tin paper with a long-tailed clip. The nanofiber membrane was placed near the nozzle of the plasma. The plasma treatment was carried out on the front and back sides of the nanofiber membrane.

3) CNT impregnation. The plasma-treated electrospun nanofiber membrane was placed in the CNT solution for 1-h ultrasonic processing, and then put into the oven at 80 ℃ for drying.

1.3 Testing and characterization

1.3.1 Morphology

The morphology of electrospun nanofiber membranes before and after CNT impregnation was evaluated by a JSM-6701F scanning electron microscope (JEOL Ltd., Japan). The voltage was 5 kV, and the current was 10 μA.

1.3.2 Nanofiber diameter

The nanofiber diameter of the electrospun nanofiber membrane was analyzed by using the ImageJ software (American National Institutes of Health, USA) according to the scanning electron microscopy (SEM) images.

1.3.3 Membrane thickness

The thickness of the electrospun nanofiber membrane was measured by an SD-201-1 thickness gauge (Dongguan Shenliang Measuring Instrument Co., Ltd., China).

1.3.4 Tensile property

The tensile properties of electrospun nanofiber membranes before and after CNT impregnation were tested by using an LT350 tensile testing platform (Linkam Scientific, UK).

1.3.5 Conductivity

The conductivity of electrospun nanofiber membranes before and after CNT impregnation was tested by using an RTS-9 four-probe tester (Guangzhou Four Probe Technology Co., Ltd., China). The parameters of the tester were set to be a square resistance with a current of 10 mA.

2 Results and Discussion

2.1 Morphology and nanofiber diameter of PAN nanofiber membranes

SEM images and the corresponding nanofiber diameter distribution of PAN nanofiber membranes are shown in Fig. 1. It can be seen that PAN nanofibers can be successfully electrospun from PAN solutions. The diameters of the nanofibers are relatively uniform and consistent. There is no beading or sticking. All nanofibers are continuous and straight, forming three-dimensional porous network structures. The diameters of nanofibers increase gradually with the increase of the PAN mass fraction in the electrospinning solution.

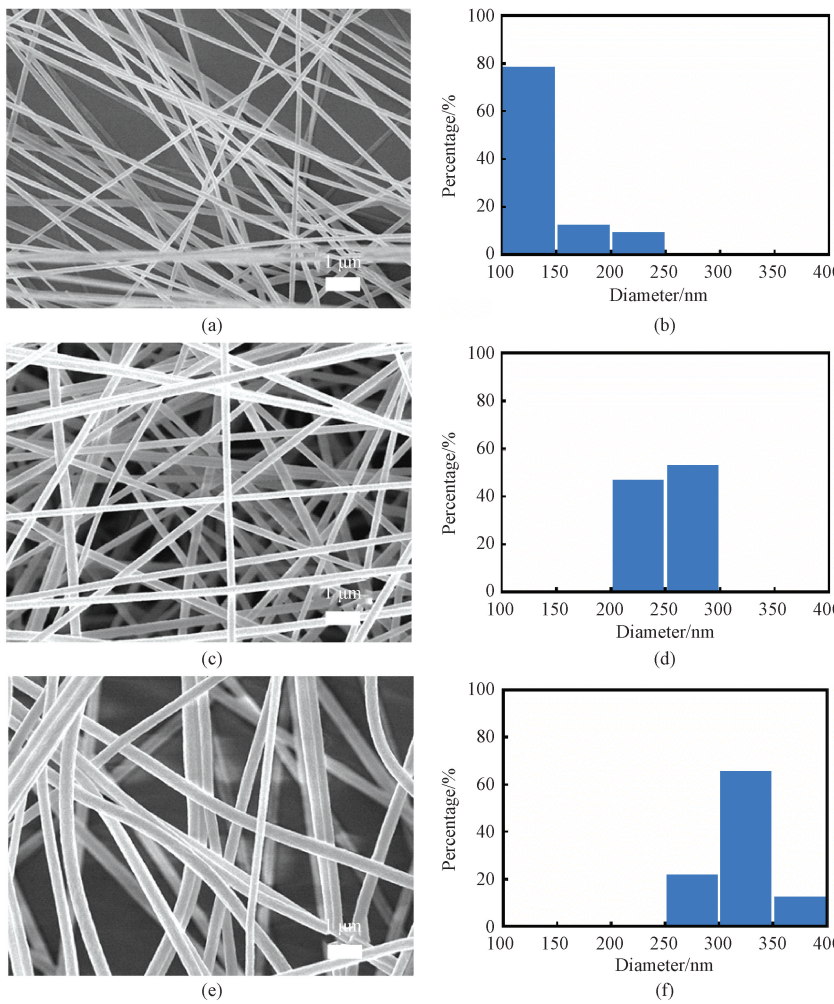


Fig. 1 SEM images and corresponding nanofiber diameter distribution of PAN nanofiber membranes at different PAN mass fractions: (a)-(b) 10%; (c)-(d) 12%; (e)-(f) 14%

The relationship between the average nanofiber diameter and the PAN mass fraction is shown in Fig. 2, where R_1^2 reflects the degree of the linear correlation between the average nanofiber diameter y_1 and the PAN mass fraction x_1. R_1^2 is close to 1, indicating a linear relationship between the average nanofiber diameter and the PAN mass fraction.

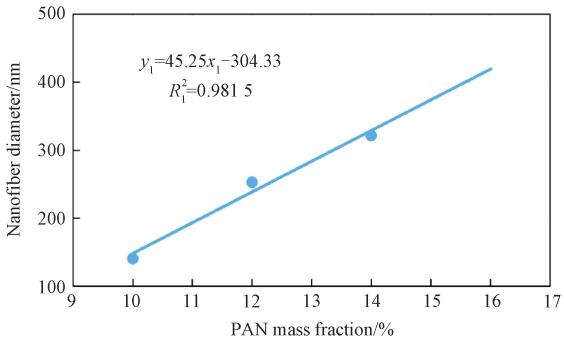


Fig. 2 Relationship between average nanofiber diameter and PAN mass fraction of PAN nanofiber membranes

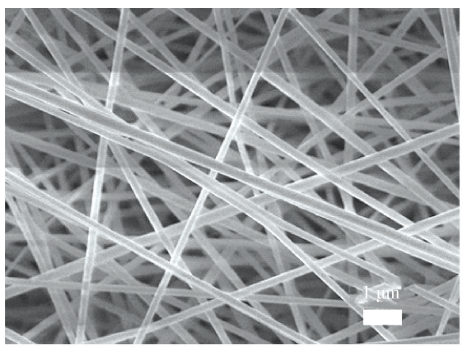
When the PAN mass fraction is 10%, 12% and 14%, the average nanofiber diameter is 141, 253 and 322 nm, respectively. The nanofiber diameter increases with the increase of the PAN mass fraction.

In theory, the tensile strength of the electrospun nanofiber membrane does not increase with the increase of the PAN mass fraction. Thicker nanofibers may increase the contact area between nanofibers, enhancing friction forces, which could improve the tensile strength of the electrospun nanofiber membrane. However, thicker nanofibers are generally less flexible and more prone to stress concentration during stretching, which could lead to a decrease in the tensile strength. Besides, the nano-reinforcement CNC would be added to the electrospinning solution. The addition of CNC greatly affects the solution viscosity and the electrospinning process. Adding a small amount of CNC would make the electrospinning process difficult when the PAN mass fraction is too high. When the PAN mass fraction is too low, CNC addition would easily lead to the poor nanofiber diameter uniformity. Thus, in the follow-up study, the PAN mass fraction is set to be 12% to electrospin the nanofiber membrane.

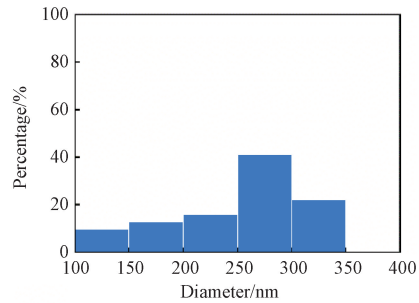
2.2 Morphology and nanofiber diameter of PAN/CNC nanofiber membranes

SEM images and the corresponding nanofiber diameter distribution of PAN/CNC nanofiber membranes are shown in Fig. 3. The nano-reinforcement CNC can be well wrapped by the PAN nanofiber. The PAN/CNC nanofibers at different CNC mass fractions are uniform, and there is no serious beading, filament or adhesion phenomenon. The nanofiber membrane also exhibits a three-dimensional porous network structure. As the CNC mass fraction increases, the difficulty of electrospinning increases.

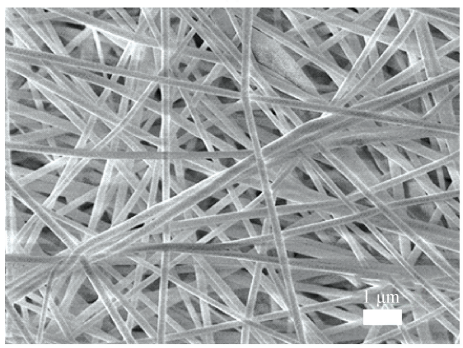
The addition of CNC could affect the nanofiber diameter. The nanofiber diameter of the PAN nanofiber membrane (a PAN mass fraction of 12%) without CNC addition is distributed in 200–300 nm. After the CNC addition, the diameter uniformity of the nanofibers becomes poor, and the nanofiber diameter distribution becomes wide. With the increase of CNC mass fraction, the nanofiber diameter has an increasing tendency.



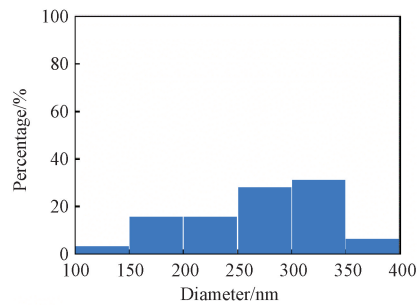
(a)



(b)



(c)



(d)

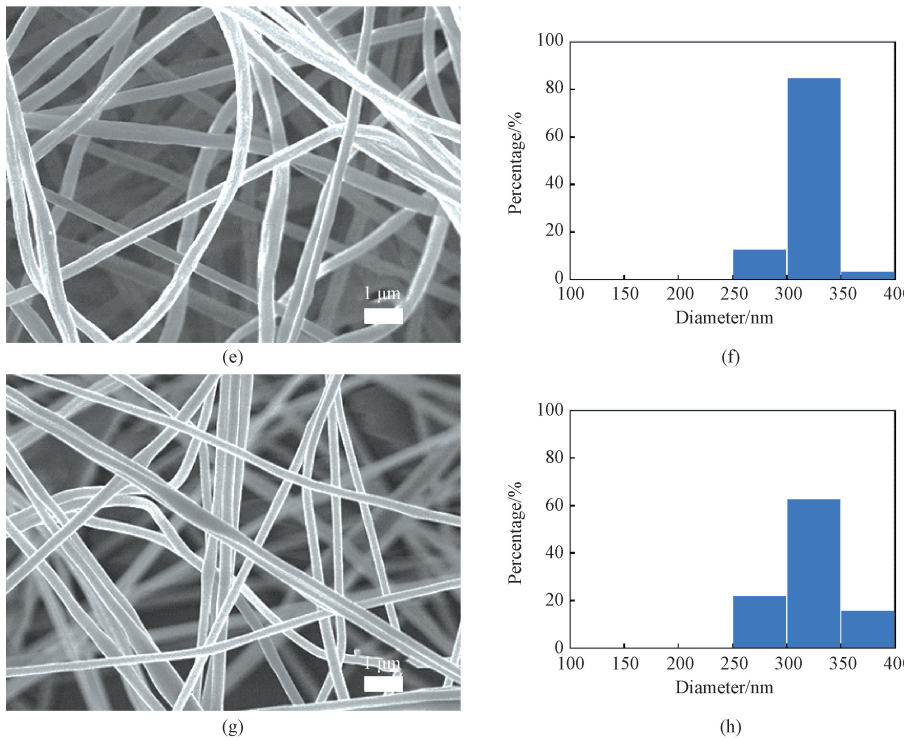


Fig. 3 SEM images and corresponding nanofiber diameter distribution of PAN/CNC nanofiber membranes at different PAN/CNC mass ratios: (a)-(b) 5∶1; (c)-(d) 4∶1; (e)-(f) 3∶1; (g)-(h) 2∶1

Table 1 shows the average nanofiber diameters of PAN/CNC nanofiber membranes. The nanofiber diameter increases after the addition of CNC. When CNC is added with a low content, there is little effect on the diameter of the nanofibers. The average nanofiber diameter is 255 nm when the mass ratio of PAN to CNC is 5∶1. The average nanofiber diameter tends to increase significantly as the mass fraction of CNC increases. This may be due to that the addition of CNC affects the properties of the electrospinning solution and thereby affects the nanofiber diameter.

Table 1 Average nanofiber diameters of PAN/CNC nanofiber membranes at different PAN/CNC mass ratios

PAN/CNC mass ratio	Average nanofiber diameter/nm
5∶1	255
4∶1	272
3∶1	316
2∶1	325

2.3 Effect of electrospinning time on thickness of PAN/CNC nanofiber membranes

With the speed, voltage and distance from the needle to the collection all being fixed, the relationship between the thickness of the PAN/CNC nanofiber membrane (a PAN mass fraction of 12% and a PAN/CNC mass ratio of 4∶1) and the electrospinning time is shown in Fig. 4, where R_2^2 reflects the degree of the linear correlation between the nanofiber membrane thickness y_2 and the electrospinning time x_2.

When the electrospinning time is shorter, the membrane is thinner. When the electrospinning time is extended from 4 h to 5 h, the thickness of the PAN/CNC nanofiber membrane increases from 0.040 mm to 0.085 mm by 112.5%. When the electrospinning time is extended from 4 h to 8 h, the thickness of the PAN/CNC nanofiber membrane increases from 0.040 mm to 0.411 mm by 927.5%. R_2^2 is close to 1, indicating an approximately linear relationship between the membrane thickness and the electrospinning time.

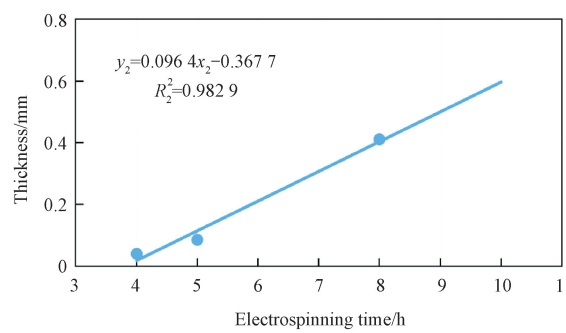


Fig. 4 Relationship between membrane thickness and electrospinning time of PAN/CNC nanofiber membranes

2.4 Morphology of PAN/CNC @ CNT nanofiber membranes

SEM images of PAN/CNC and PAN/CNC @ CNT nanofiber membranes at a PAN/CNC mass ratio of 4∶1

are shown in Fig. 5. It is evident that CNTs can be effectively loaded onto the electrospun nanofiber membrane. CNTs contribute to the stable conductive network, and improve the conductive performance.

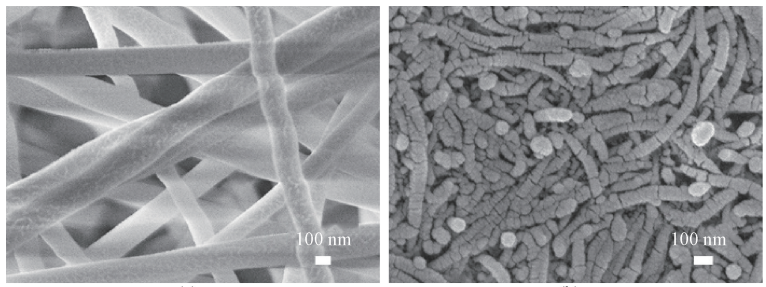


Fig. 5 SEM images of nanofiber membranes: (a) PAN/CNC; (b) PAN/CNC@CNT

2.5 Tensile properties of PAN, PAN/CNC and PAN/CNC@CNT nanofiber membranes

The tensile stress-strain curves of PAN, PAN/CNC and PAN/CNC@CNT nanofiber membranes are shown in Fig. 6. The tensile strength of the PAN nanofiber membrane is 0.81 MPa. The addition of CNC affects the tensile properties of the PAN nanofiber membrane. With the increase of the CNC mass fraction, the tensile strength of the PAN/CNC nanofiber membrane first increases and then decreases. When the mass ratio of PAN to CNC is 4∶1, the tensile strength reaches the maximum, which is 2.13 MPa. The result shows that the addition of CNC could enhance the tensile strength of nanofiber membranes. The strong interfacial interaction between CNC and the compatible polymer results from hydrogen bonding and enables the effective stress/load transfer between the reinforcement and the matrix, resulting in high stretching resistance as well as high performance of nanocomposites[26, 30]. The tensile strength of the PAN/CNC@CNT nanofiber membrane at a PAN/CNC mass ratio of 4∶1 increases to 3.12 MPa. Compared with that of the PAN nanofiber membrane, the tensile strength of the PAN/CNC@CNT nanofiber membrane increases by about 285%. This indicates that CNTs can further improve the tensile properties of PAN/CNC nanofiber membranes due to the high elastic modulus and tensile strength of CNTs.

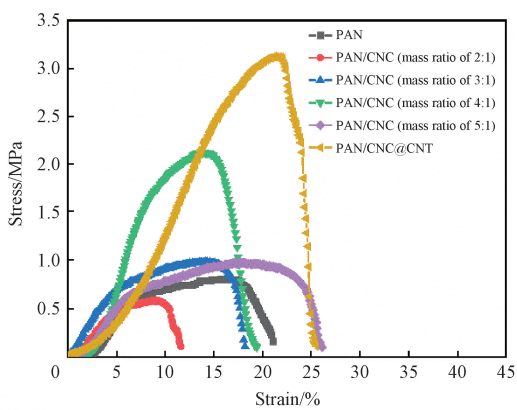


Fig. 6 Tensile stress-strain curves of electrospun nanofiber membranes

2.6 Conductivity of PAN/CNC and PAN/CNC@CNT nanofiber membranes

The surface resistivity of PAN/CNC and PAN/CNC@CNT nanofiber membranes at a PAN/CNC mass ratio of 4∶1 is shown in Table 2. PAN is an insulating material, and CNC is also an insulating or semiconducting material. The PAN/CNC nanofiber membrane has a very low conductivity and it may not be measurable by the four-point probe method. After CNT impregnation, the surface resistivity changes to 64 Ω/cm^2. This is because the surface of the PAN/CNC nanofiber membrane is loaded with a certain amount of CNTs which have a high conductivity. Therefore, the flexible PAN/CNC@CNT nanofiber membrane has a higher conductivity.

Table 2 Surface resistivity of PAN/CNC and PAN/CNC@CNT nanofiber membranes

Nanofiber membrane	Surface resistivity/(Ω/cm^2)
PAN/CNC	—
PAN/CNC@CNT	64

3 Conclusions

CNC reinforced PAN nanofiber membranes were successfully prepared by electrospinning. Flexible conductive PAN/CNC@CNT nanofiber membranes were successfully prepared by impregnating PAN/CNC nanofiber membranes with CNTs. With the increase of the PAN mass fraction, the viscosity of the electrospinning solution increases and the nanofiber diameter becomes larger. The PAN/CNC nanofiber membranes can be successfully prepared with the addition of nano-reinforcement CNC at a PAN mass fraction of 12%. The addition of CNC affects the structure and properties of electrospun nanofiber membranes. With the addition of CNC, the nanofiber diameter increases and the nanofiber diameter distribution widens. When the mass ratio of PAN to CNC is 4∶1, the tensile strength of the PAN/CNC nanofiber membrane is the highest, and it is higher than that of the PAN nanofiber membrane. After CNT impregnation, the tensile strength of the flexible conductive PAN/CNC@CNT nanofiber membrane

increases from 2.13 MPa to 3.12 MPa, and the surface resistivity changes to 64 Ω/cm^2. The flexible conductive nanofiber membranes could be used in energy storage and sensing fields, and this study might provide a strong base for their future development.

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纤维素纳米晶提高柔性导电聚丙烯腈@碳纳米管纳米纤维膜的拉伸性能

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摘　要：柔性导电纳米纤维膜在可穿戴电子设备领域的应用非常广泛，而良好的拉伸性能是静电纺纳米纤维膜商业化的必备性能之一。以纤维素纳米晶（cellulose nanocrystal，CNC）为增强材料，通过对含 CNC 的 PAN 溶液的静电纺丝及碳纳米管（carbon nanotube，CNT）水分散液的浸渍处理，成功制备了柔性导电聚丙烯腈（polyacrylonitrile，PAN）/CNC@CNT 纳米纤维膜。对该纳米纤维膜的结构和性能进行研究。结果表明：随着纺丝液中 PAN 质量分数的增加，纺丝液的黏度增加，纤维直径增加。当 PAN 质量分数为 12%时，可成功制备不同 CNC 质量分数的 PAN/CNC 纳米纤维膜。CNC 的添加对 PAN/CNC 纳米纤维膜的结构和性能产生了影响。随着 CNC 质量分数的增加，纤维直径增加，直径分布范围变宽，拉伸强度先提高后降低。当 PAN 与 CNC 的质量比为 4∶1时，PAN/CNC 纳米纤维膜的拉伸强度最高，明显高于 PAN 纳米纤维膜。经过 CNT 水分散液浸渍处理得到的 PAN/CNC@CNT 纳米纤维膜的拉伸强度增加至 3.12 MPa，表面电阻率为 64 Ω/cm^2。柔性导电纳米纤维膜有望广泛应用于储能、传感等领域，该研究结果可为上述领域的研究提供一定的基础。

关键词：纤维素纳米晶；柔性导电纳米纤维膜；拉伸性能；碳纳米管；静电纺丝；聚丙烯腈