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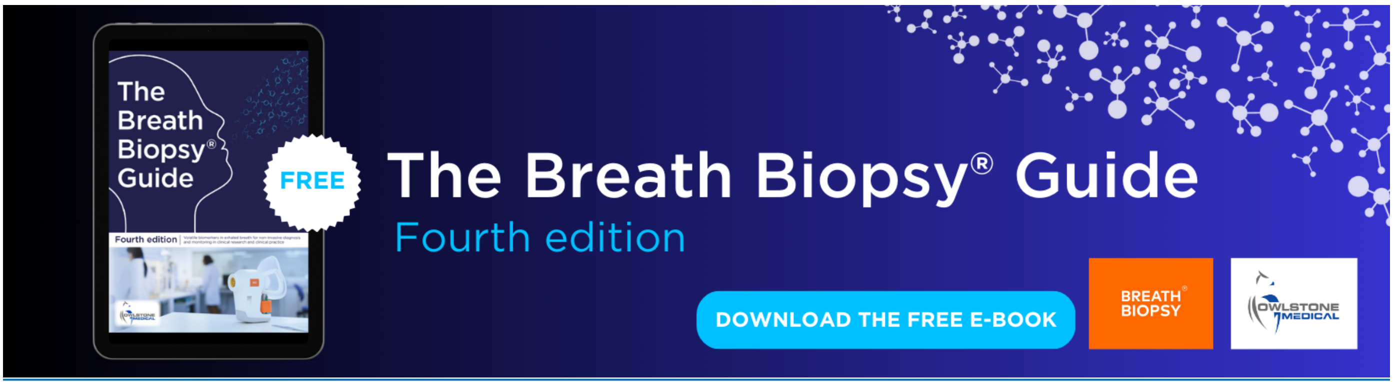

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Investigation on the mechanical properties of composite fiber membranes with different concentration of EVOH/Ag/AC

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Keywords: electrospinning, EVOH, AgNPs/AC, tensile test, mechanical properties

Abstract

There are several requirements of properties for filter material: adsorption, antibacterial property, air permeability, mechanical properties, etc. Electrospinning technology is a recognized method with great potential to fabricate nanofiber membrane. In particular, the mechanical properties need to be well understood for applications where the membrane is subjected external loading, e.g. tension forces which could be monotonic or cyclic. In this study, the mechanical properties of ethylene vinyl alcohol (EVOH) nanofiber membranes loaded with silver nanoparticles (AgNPs) and activated carbon (AC) were investigated in detail. Static load tensile tests were carried out for nine groups of membranes with different mass fraction of EVOH (7.5%, 10%, 12.5%) and concentration ratios of Ag/AC (2:5, 1:1, 5:2). The results showed that the tensile strength and Young's modulus of this membrane would increase with the increase of EVOH mass fraction. When the mass fraction of EVOH is 7.5% and the concentration ratio of Ag/AC is 1:1, the fracture strength of nanofiber membrane was the highest, which was 0.1545 MPa.

1. Introduction

The long-term emissions of harmful particles, industrial wastes and toxic gases have caused severe air and water pollutions. These will increase the likelihood of people suffering from respiratory and heart diseases, even lead to chronic or acute poisoning which seriously affect health and life [1, 2]. Therefore, it is necessary to filter the toxic chemicals as much as possible before emitting them. Nano filter materials fabricated by electrospinning technology are promising filter materials because of the large specific surface area, high porosity, small aperture and other merits [3, 4]. Figure 1 shows the simple schematic of the electrospinning work area. The high voltage is applied between the collector and the syringe which contains the polymer melt or solution. Under high voltage field, the electrified jet of polymers is generated near the tip and split to micron- to nano- scaled fibers [5]. Since the charges of the collector and the fibers are opposite, the fibers will be attracted to the collector. In addition, there is always a distance between the collector and the tip to some extent, the fibers are solidified and stretched to develop nano-fibers [6]. A fibrous membrane is then gradually developed by collecting lots of fibers. Luo *et al* [7] fabricated the polyvinylidene difluoride (PVDF)/polystyrene (PS) films by electrospinning for the filter layer of the mask. It was proved that the PVDF/PS film could filter air particles with all diameters and its filtration performance can be recovered with friction. Deng *et al* [8] manufactured the Sodium sulphobutylether-β-cyclodextrin (SBE-βCD)/Polyvinyl alcohol (PVA) film with multi-hierarchical loose structure by electrospinning technique. This product performed a high-efficiency, breathable and sustainable air filtration effect. Beatriz *et al* [9] processed the poly (ethylene-co-vinyl alcohol) (EVOH) film by electrospinning assisted by the coaxial technology. They found that this nanocomposite of enhanced rigidity was obtained with the incorporation of hydrolytically extracted cellulose nanocrystals (CNCs). In addition, with the further researches,

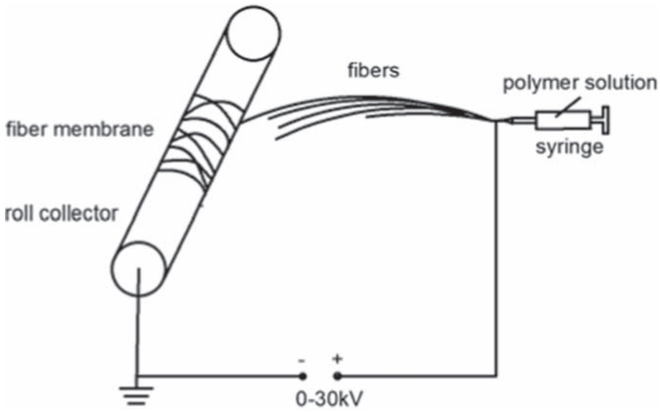

Figure 1. Work area of electrospinning.

particles with adsorption and antibacterial properties were considered to load on the nanofiber for better performance [10, 11]. Dan *et al* [12] fabricated PVA/konjac glucomannan (KGM)-based nanofiber membranes with ZnO nanoparticles by electrospinning and ecofriendly thermal cross-linking. The results showed that the membranes performed great antibacterial ability and high removal efficiency for air filtration. Sri *et al* [13] synthesized the polyacrylonitrile (PAN)/TiO_2/Ag nanofibers membrane by electrospinning technique for air filtration. They demonstrated that there was hydrophilic surface of the film with the addition of TiO_2 and Ag and its highest efficiency for PM2.5 reached 96.9%. Yang *et al* [14] prepared the PVA/PAN/activated carbon fibers with electrospinning technology which had a better air particle filtration efficiency.

In this study, ethylene vinyl alcohol (EVOH) nanofiber membrane loaded with silver nanoparticles (AgNPs) and activated carbon (AC) was fabricated by electrospinning for better performance of filtration. The reason that EVOH was selected as the basic material for the nanofiber scaffold is due to its good mechanical strength, elasticity and biodegradability [15] which is a better choice for packaging, medical, film materials, etc. The adsorption and antibacterial properties of AgNPs-AC EVOH composite fiber membrane have been verified in previous study [16]. However, the mechanical properties of a membrane should also be considered as a standard for evaluating practical application [17]. Itoh *et al* [18] manufactured PVA nanofibers by free surface electrospinning with the solutions of different conductivity. The results indicated that the nanofibers mats were obtained with higher tensile strength due to the formation of ribbon-shaped fibers. Pham *et al* [19] investigated the influence of fiber orientation on the mechanical properties of polyvinyl chloride (PVC) nanofiber mats by changing the drum collector's rotation speed. The results showed that the mechanical properties of the mats were improved when the speed increased from 0 to 2500 rpm.

The amelioration of mechanical properties of fiber membranes can extend their service life and improve the efficiency of filtration. From others' work above, the influence of processing parameters (such as fibers' orientation, the applied voltage, the diameters of fibers, etc) on mechanical properties has been investigated [20]. However, for this new composite fiber with good adsorption and antibacterial properties, the impact of mixed additives (AgNPs/AC) on the morphology and mechanical properties of the membrane is still unclear. With the premise of better adsorption and antibacterial properties, the obtained membrane is supposed to have better mechanical properties. The main work of this study was to evaluate their mechanical properties for providing a reference for selecting appropriate raw material proportions under different performance requirements.

It is known that the proportion of raw materials affected the adsorption and antibacterial properties by influencing the morphology of the membrane [16, 21]. Similarly, the mechanical properties also mainly depend on it. Therefore, the primary content of this work is to investigate the mechanical properties of membranes with different proportion of raw material, i.e., EVOH, AgNPs and AC. According to the previous study, the mass fractions of EVOH were set as 7.5%, 10%, 12.5% and the different Ag/AC ratios were chosen as 2:5, 1:1 and 5:2. Finally, nine groups of composite membranes were fabricated. After analyzing their morphology, the static load tensile tests were carried out on these membranes. The mechanical results are analyzed and compared thoroughly in order to provide a reference for the selection of electrospinning raw materials.

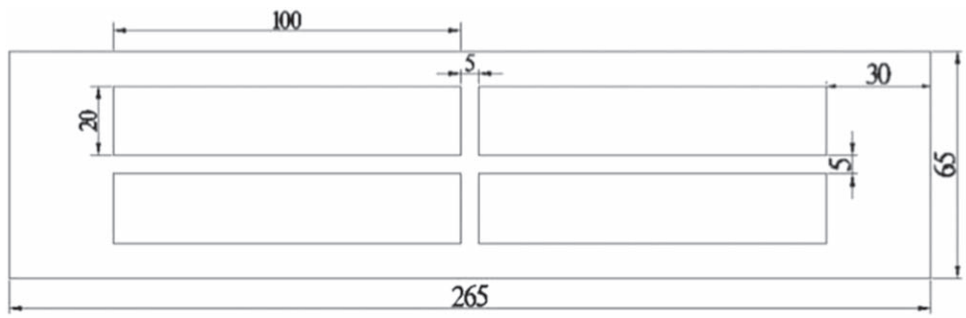

Figure 2. Parallel sampling method (dimensions are in mm).

2. Experiments

2.1. Fabrication of Ag/AC EVOH composite fiber membranes

The EVOH solid particles were dissolved in a mixture of isopropyl alcohol and deionized water (7:3) with heating in water bath (75 °C) and stirring by magneton. After EVOH was completely dissolved, silver nitrate solid and powdered activated carbon were added successively till fully dissolved and evenly mixed. The polymeric precursor solution was thus prepared well.

The electrospinning equipment (SS-3556H, Beijing TECHNOVA Technology CO., LTD) was used in this study. The fabrication parameters were settled as follows: needle's diameter was 0.6 mm, voltage was 17 kV, the distance between needle and roll receiver was 17 cm, injection velocity was 0.2 mm min^{-1}, temperature was 40 °C. Finally, nine groups of nanofiber membranes were fabricated with different mass fraction of EVOH (7.5%,10%,12.5%) and different Ag/AC ratios (2:5, 1:1, 5:2).

2.2. Tensile tests

For testing the mechanical properties of nanofiber membrane, the results are heavily influenced by the sampling method. The nanofibers obtained by electrospinning are mainly collected to the receiver in the form of spiral, the thickness distribution of the obtained membrane is thus not uniform. Besides, the fibers are directional to some extent inside a membrane resulting from the roll receiver. Therefore, it is important to select a suitable sampling method for tensile test of nanofiber membrane. A parallel method is the most suitable sampling method for electrospinning nanofiber film [22]. In this study, four parallel 2 cm × 10 cm sample strips were taken from the center of the fiber membrane where the thickness was more uniform. The specific specification is shown in figure 2.

The tensile properties of fiber mainly include yield stress, tensile modulus, fracture strength and yield strain. An electronic universal testing machine (CMT4503) was used for the static load tensile test. This machine adopts a uniquely designed mechanical sensor, combined with a capacitive displacement sensor, to allow the system to load the sample accurately. The maximum load of this machine is 100 N, besides, the nominal accuracy is 0.1 grade and the load resolution is 333 uN. The fiber membrane sample is placed between the upper and lower jigs, in which only one jigs moved freely. The tensile tests were carried out based on the standard GB/T 3354-2014 [23]. The gauge was 60 mm and the stretching velocity was 2 mm min^{-1}.

3. Experimental results and discussions

3.1. Morphology observations on membranes

The morphology observations of membranes' structures are conducive to better understand and analyze the macroscopic mechanical results from the microstructure. Figure 3 shows the SEM images of the obtained membranes with different mass fraction of EVOH but the same concentration ratio of Ag/AC of 1:1. It is obvious that the diameters of fibers are increase with the increase of mass fraction of EVOH, which should be due to the higher viscosity. Besides, it can be seen that the amount of fibers in figure 3(a) are much smaller than those in figures 3(b) and (c). Of course, the artificial selection of observation samples is a possible reason for this result. Another reason might be that the distribution of fibers on the roller is more dispersed resulting from the lighter weight of fibers in figure 3(a). However, the overall orientation of the fibers in figure 3(a) is better than that of others.

In addition, it can be observed that the size of particles attached to the fibers in figure 3(c) is generally larger than that in figure 3(a). The larger particles are mostly at the knots of multiple fibers, increasing the friction

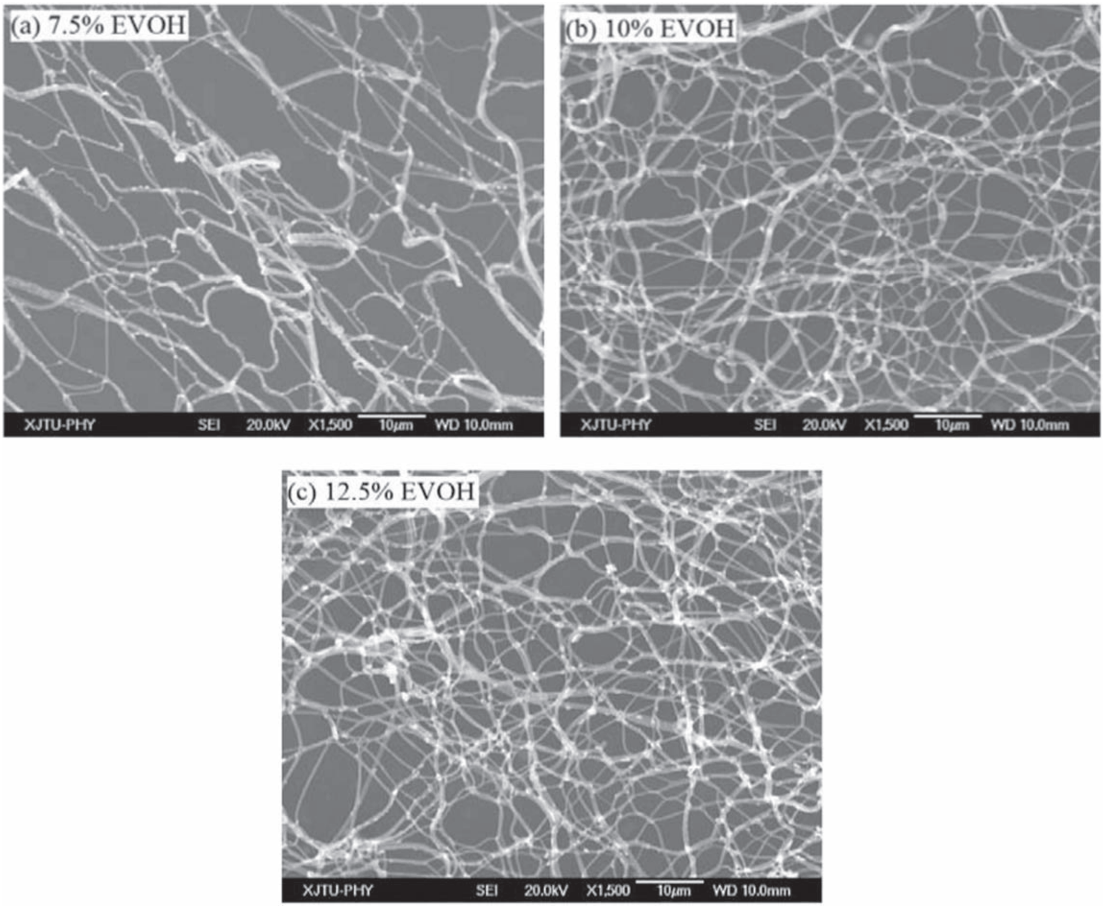

Figure 3. SEM images of membranes with AgNPs/AC ratio of 1:1 and different mass fraction of EVOH of (a) 7.5% (b)10% (c) 12.5%.

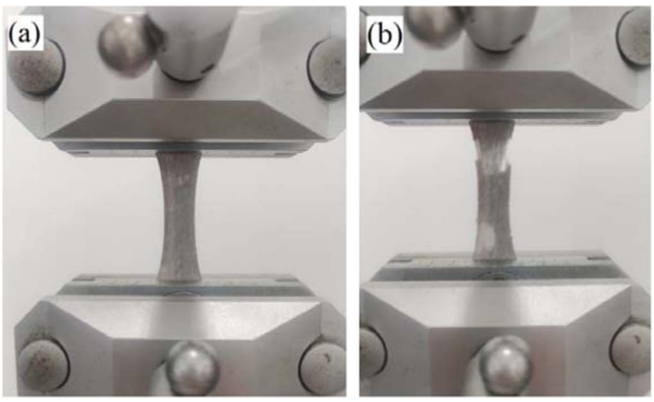

Figure 4. Tensile test phenomena (a) necking (b) fracture.

between fibers during the stretching process. Therefore, in the perspective of microstructure, it is assumed that the mechanical properties will be better when the mass fraction of EVOH is 12.5%.

3.2. Physical phenomenon of tensile test

Figure 4 shows the phenomena during the tensile test, including (a) necking and (b) fracture. The necking occurred at the beginning of stretching resulting from many individual fibers inside the membrane. From the perspective of the microscope, the external force loaded on each single fiber at the same time when the fiber membrane is stretched. The direction of the tension on each fiber is basically different from its own direction. As the tension increases, the direction of each fiber changes in the direction of the force. Uneven force thus

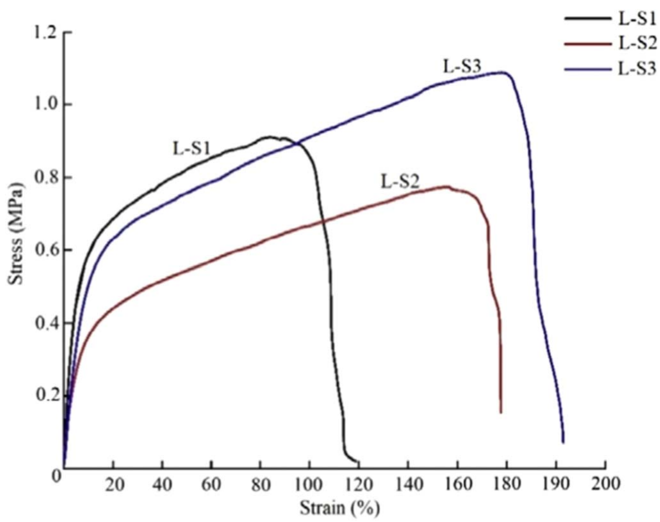

Figure 5. Stress–strain curve of membrane with EVOH of 7.5%.

appeared on the surface of the membrane resulting in different degrees of necking. When the tension is further increased, the fibers gradually break.

The fracture of fiber membrane is a continuous process from micro to macro, which is complicated. Three stages can be observed during the tensile test of the frber membrane from the beginning of tensile stretch to the break: elastic stage, plastic stage and fracture failure stage [24, 25].

At elastic stage, due to the small external force on the fiber, the main occurrence is the deformation of the macromolecular chain itself in the fiber due to the change of bond length and bond angle. Stretching at this stage is called the Hooke region, expressing the approximately linear curve. The molecular chain and transverse connection bond of the fiber return to the original position after removing the force, which is a complete elastic recovery.

At plastic stage, the external force on the fiber increases. The macromolecular chains in the non-crystalline zone overcome the secondary valence bond force between the molecular chains, casuing the subsequent stretching and orientation. Some of the macromolecular chains are straightened while some transverse connection bonds are broken. The dislocation slip between the macromolecules in the non-crystalline zone is gradually developed because of the fracture of the secondary valence bond. Therefore, it is easier to lengthen the fiber. Nevertheless, the stress rises very slowly and the slope of the stress–strain curve is small.

At the fracture stage, the external force on the fiber increases further. The distance between macromolecules becomes smaller after plastic deformation, a new secondary bond is formed between the molecular chains. With continuous loading, the fiber deformation is then mainly caused by the stretching of the macromolecular chain itself and the destruction of the secondary bond. The stress increases rapidly, resulting in a strengthening phenomenon. Fiber fracture finally occurs with the break of the fiber macromolecular backbone and most secondary bonds.

3.3. Mechanical properties of membranes with different concentration ratios of Ag/AC

Figure 5 shows the stress–strain curves of the membrane with EVOH of lower mass fraction of 7.5% and concentration ratios of Ag/AC of 2:5, 1:1 and 5:2, which were labelled as L-S1, L-S2 and L-S3, respectively. It can be seen that the membrane represented by L-S1 broke first, which indicated the smallest breaking strength and small tensile strength of this membrane [26]. For L-S2, the maximum stress value was the smallest, demonstrating the smallest tensile strength, but high fracture strength. When the proportion of silver concentration was higher (corresponding to L-S3 in figure 5), this membrane was the last one to break, indicating the maximum fracture strength and high tensile strength.

When the mass fraction of EVOH is the medium level of 10%, the stress–strain curves of membranes with concentration ratios of Ag/AC of 2:5, 1:1 and 5:2 are shown in figure 6, which were labelled as M-S1, M-S2 and M-S3, respectively. If the concentration of AC was higher, the tensile strength is smaller and the fracture strength was the smallest. When the silver/activated carbon concentration ratio is 1:1, the tensile strength of the corresponded membrane was the smallest and the fracture strength was also small. From the M-S3 curve, the tensile strength and fracture stress of the membrane with higher Ag concentration were both largest.

Figure 7 depicts the stress–strain curves of the membranes with EVOH of higher mass fraction of 12.5% and concentration ratios of Ag/AC of 2:5, 1:1 and 5:2, which were labelled as H-S1, H-S2 and H-S3, respectively. The

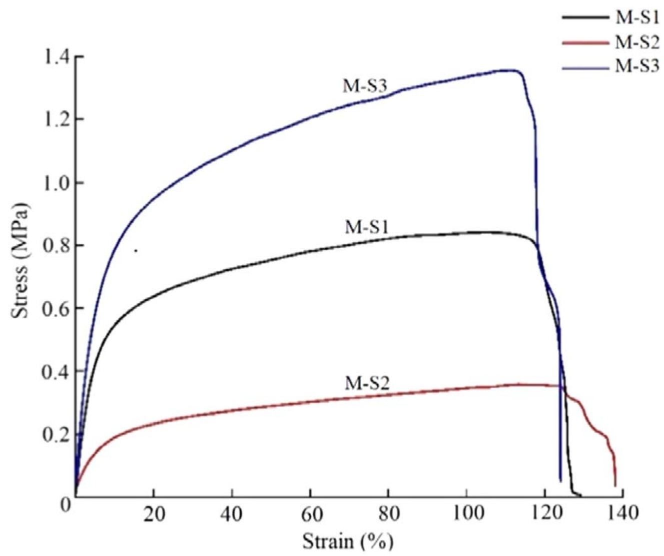

Figure 6. Stress–strain curves of membranes with EVOH of 10%.

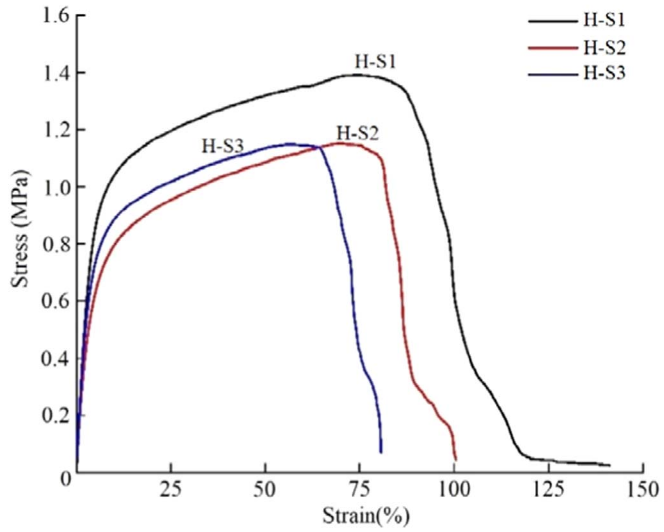

Figure 7. Stress–strain curve of membrane with EVOH of 12.5%.

tensile strength is the largest and the fracture strength was the smallest when the concentration of AC was higher. As the membrane with the Ag/AC concentration ratio of 1:1, both tensile strength and fracture strength were higher. When the proportion of silver concentration is higher, the tensile strength of the membrane was the smallest while the fracture strength was largest.

In summary, it was found that the mechanical properties of Ag/AC composite fiber membrane were affected by the high solute properties. At the initial stage of stretching, the stress of the membranes increased sharply with the linear relationship of strain. When the strain was about 8%, the fiber entered the plastic deformation stage. The stress increased slowly in a non-linear relationship with the strain. From figures 5–7, it can be seen that the fracture strains were between 75% and 200%.

3.4. Yield strength analysis of membranes with different concentration ratios of Ag/AC

Compared with other metal and non-metal materials, the yield deformation of nanofiber materials is different. On the one hand, there is no obvious yield deformation of nanofiber material because of its intrinsic features [27]. On the other hand, there are great differences on the yield deformation of nanofibers with different concentrations, besides, the yield deformation occurred at different times [28]. Therefore, for the solution of the yield strength of the membranes, the stress value corresponding to the overall deformation of 0.2% was taken as the yield strength, which is convenient to compare the tensile properties of different membranes [29]. Figure 8

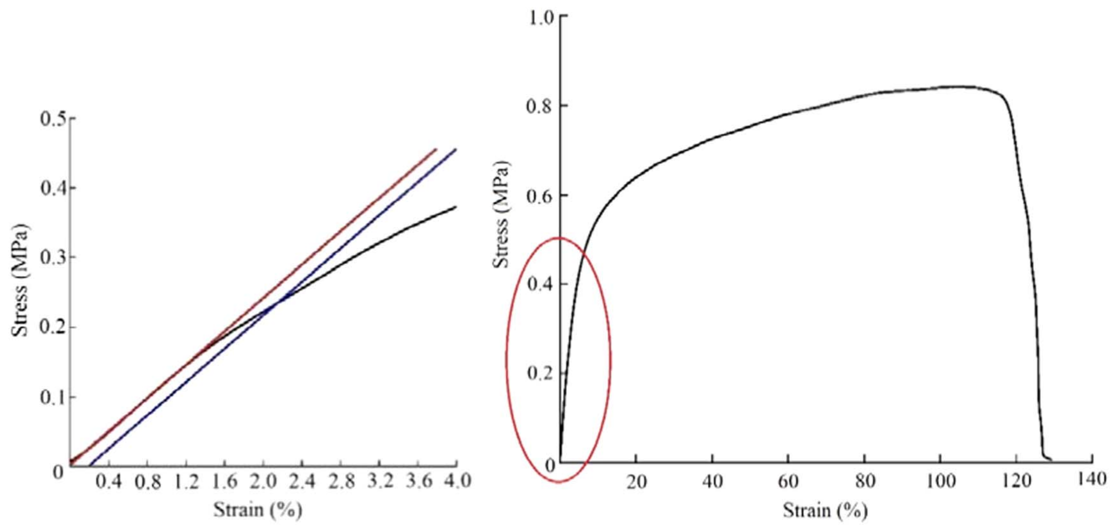

Figure 8. Solutions of Young's modulus and yield strength.

demonstrates the solution process of Young's modulus and yield strength. That is, the tangent line of the curve segment at the point where the deformation is 0 was calculated in the part of each curve whose deformation is in the range of 0%–4%. The value of Young's modulus was represented by the slope of the tangent line. The tangent line was then translated until the strain value was 0.2%. In this case, the ordinate value of the intersection point between the translation line of the tangent line and the curve was the yield strength value.

The Ag/AC composite fiber membrane showed a yield state at a strain range of about 0%–2%. With the further stretch, the maximum strain reached 205%. In addition, the membrane with higher concentration of AC showed a larger yield strength.

The tensile properties of nanofiber films are obtained according to the stress–strain curves of tested Ag/AC composite fiber membranes. Figure 9 shows the values of four mechanical properties change with the different mass fraction of EVOH and concentration ratios of Ag/AC. It can be found that the maximum value of tensile strength of Ag/AC composite fiber membrane was 1.3919 MPa, as the concentration ratio of Ag/AC was 2:5. Besides, the Young's modulus and yield strength were also the maximum values, which were 0.2562 MPa and 0.5956 MPa, respectively. The fracture strength of the composite membranes reached the maximum value of 0.1545 MPa when the concentration ratio of Ag/AC was 1:1, however, the tensile strength was only 0.7745 MPa.

Overall, when the concentration ratio of Ag/AC is 5:2, the mechanical properties of the membrane are better. At the same concentration ratio of Ag/AC, the mechanical properties of membrane are better when the mass fraction of EVOH is 12.5%, which is basically consistent with the morphology observation.

3.5. Mechanical properties of the membranes with different mass fraction of EVOH

By analysis of the mechanical properties of these membranes above, it can be concluded that the membrane performed better mechanical properties when the concentration ratio of Ag/AC was 5:2. This indicated that the concentration of AC has a higher influence on the mechanical properties. Therefore, with the Ag/AC concentration ratio of 5:2, the influence of different mass fraction EVOH on the mechanical properties of membranes is analyzed, as shown in figure 10.

From figure 10, it is found that when the strain range is 0%–40%, the stress value increased with the increase of EVOH mass fraction under the same strain condition. It was mainly due to the bigger diameter of electrospun nanofibers with the increase of polymer mass fraction [30]. Therefore, the better rigidity of the membrane lead to the reduction of the elastic deformation. When the EVOH mass fraction was 12.5%, the fracture strength, yield strength and Young's modulus of the nanofibers were greater than those with EVOH mass fraction of 7.5% and 10%, which were 0.076 MPa, 0.2351 MPa and 0.5861 MPa, respectively.

In addition, it can be seen from figure 10 that the membrane with a large EVOH mass fraction broke first, but with higher tensile strength. This illustrated that the fiber orientation decreased and the fiber diameter distribution was uneven with the increase of polymer content. This may be because of the low volatility of the solvent and the low ambient temperature, which caused the fiber to be insufficiently stretched during electrospinning [31].

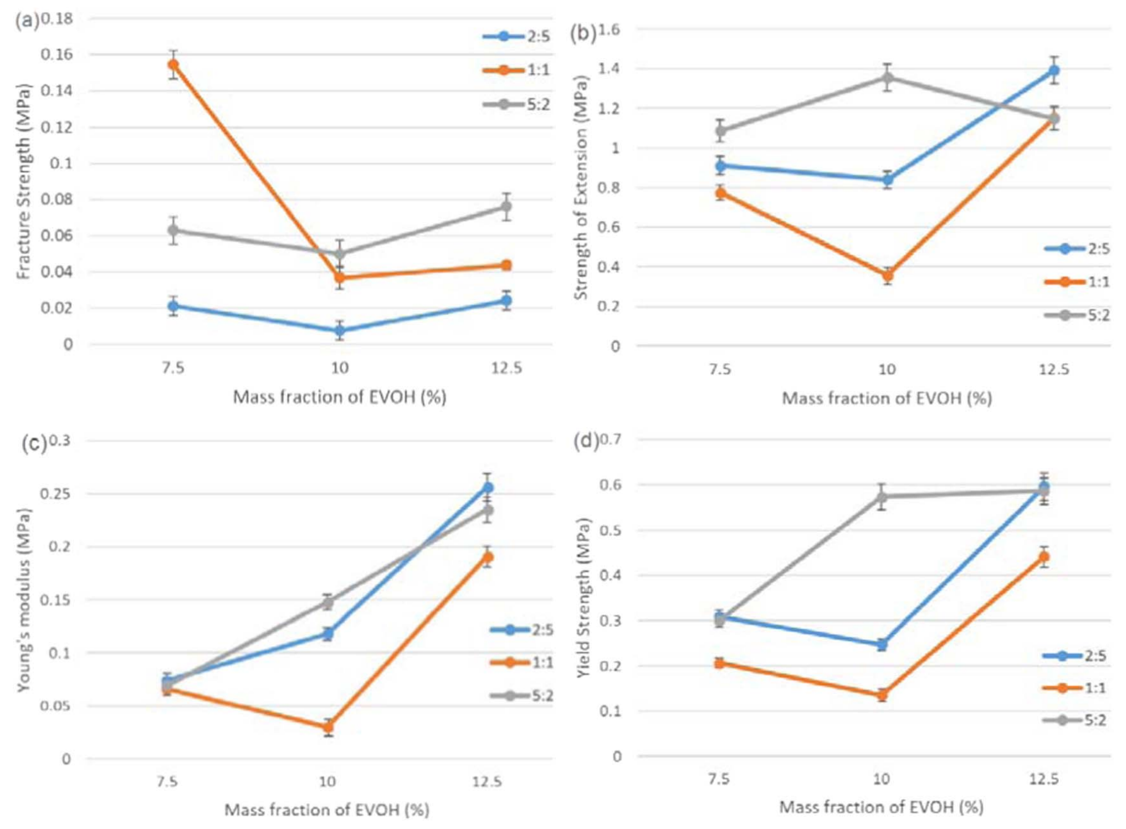

Figure 9. Tensile properties of membranes (a) Fracture strength (b) Strength of extension (c) Young's modulus (d) Yield strength.

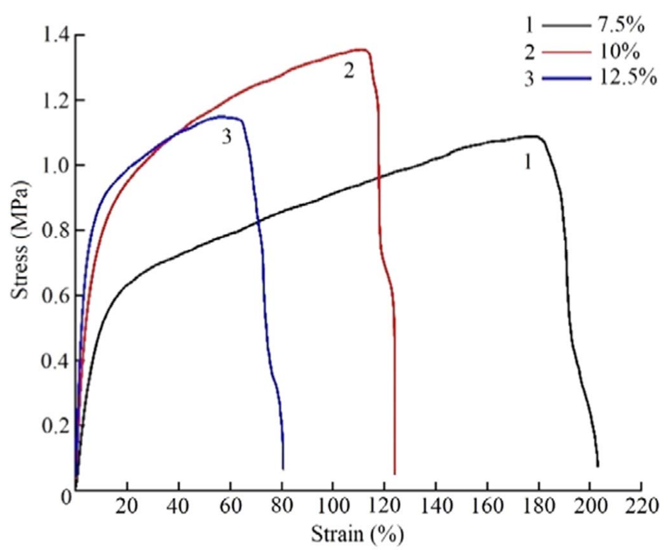

Figure 10. Mechanical properties of nanofiber membrane with different mass fractions of EVOH (Ag/AC ratio 5:2).

4. Conclusion

In this study, the mechanical properties of the composite membranes with different mass fraction of EVOH (7.5%, 10% and 12.5%) and concentration ratios of Ag/AC (2:5, 1:1 and 5:2) were tested in detail. The conclusions are summarized as below:

(1) When the mass fraction of EVOH was 7.5% and the concentration ratio of Ag/AC was 1:1, the fracture strength of the nanofiber membrane was the maximum, reaching 0.1545 MPa. When the mass fraction of

EVOH is 12.5% and the concentration ratio of Ag/AC was 2:5, the Young's modulus and yield strength of the nanofiber membrane were the highest.

(2) With the increase of EVOH mass fraction from 7.5% to 12.5%, the average tensile strength of the three membranes with different AgNPs/AC ratios increased from 0.0699 MPa to 0.2274 MPa with the growth rate of 225.32%, while that of yield stress increased from 0.2719 MPa to 0.5410 MPa with the growth rate of 98.97%.

(3) When the Ag/AC concentration ratio is 5:2, the mechanical properties of the membrane were improved.

In addition to the composition of polymer solution, the mechanical properties of electrospun fiber membrane are also affected by changing the receiving method, post-treatment, etc. The further researches will be carried out in the future.

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Data availability statement

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

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