

# Properties of Hydrophilic Chitosan/Polysulfone Nanofibrous Filtration Membrane

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## ABSTRACT

Electrospun nanofibrous membranes are useful water filtration materials due to their high interconnected porosity and tunable pore sizes, which cause very high permeability and selectivity. However, poor mechanical properties and easy fouling due to their extremely high surface area limit their applications. Therefore, it is desirable to enhance the mechanical properties and the hydrophilicity of such electrospun nanofibrous membranes. In this paper, electrospun polysulfone (PSF) nanofibrous membranes were treated with plasma. Crosslinked chitosan solution was then employed to pad the membranes. We studied the influence of the chitosan concentration and the volume of glutaraldehyde on the morphology, porosity structure, mechanical properties and hydrophilicity of electrospun polysulfone nanofibrous membranes. The results showed that the average pore size decreased from 4.5  $\mu\text{m}$  to 2.68  $\mu\text{m}$ , the breaking stress increased from 6.01 $\pm$ 0.44 MPa to 9.25 $\pm$ 0.45 MPa, and the water contact angle decreased from 130.8 $^\circ$  to 0 $^\circ$  in 30 s when chitosan was applied to the membranes. These changes occurred by padding due to the crosslinked chitosan solution. The results indicate that a significant improvement occurred in the mechanical properties; the highly hydrophobic PSF membrane was changed to a superhydrophilic one and the pore size was reduced. These results encouraged us to propose this material as a water filtration membrane with longer life span, lower fouling and higher rejection efficiency.

**Keywords:** Electrospinning; polysulfone; chitosan; mechanical properties; hydrophilicity

## INTRODUCTION

Electrospun nanofibrous membranes are used as water filtration materials because of their extraordinary permeability and strong selectivity, which result from their great porosity, tunable pore size and high surface area [1]. However, electrospun nanofibrous membranes have low mechanical strength due to their loose structure and low interactive binding force between the fibers. The properties that are vital for a water filtration material are excellent chemistry stability and fouling resistance under a specific required pressure. On the other hand, higher porosity, surface area and hydrophobicity will lead to severe occlusion of pores in the electrospun nanofibrous membranes. Therefore, to preserve the high permeability and rejection efficiency of a nanofibrous membrane, it should be mechanically reinforced and hydrophilized. Ben Chu's group [2] first made a PAN/chitosan composite electrospun nanofibrous membrane that used electrospun PAN nanofibers as the scaffold and chitosan as the top ultrathin barrier layer. For water-oil solution as the feeding liquid, the nanofibrous composite membranes produced an excellent flux rate at a high rejection efficiency (above 99%). Many modifications were then performed, such as coating and crosslinking PVA and cellulose acetate on the PAN nanofibrous membranes[3,4], and surface polymerization of polyamide on the PAN electrospun nanofibrous scaffolds[5]. Jianfen Zheng et al.[6] obtained a three-layered composite membrane composed of chitosan membrane, PVDF nanofibers and nonwoven PET, whose rejection efficiency to bovine serum albumin was up to 98.9%.

Chitosan, poly  $\beta$ -(1,4)-2-amino-2-deoxy-D-glucose], has good hydrophilicity, antibacterial and antifouling abilities. However, it swells significantly in water, making it imperfect for some filtration environments. Recently, many researchers have blended crosslinking agents into chitosan solution to form more stable structures. Some examples of such agents include GA[7,8], lactic acid[9], maleic anhydride [10], etc. The research results showed that water swelling was decreased significantly after modification by crosslinking agents and that protein adsorption was decreased because electric charges were normalized, which reduced the interaction between the chitosan and protein. Therefore, crosslinked chitosan can be used effectively in a strong acid environment.

Polysulfone (PSF) is one of the most commonly used water filtration materials due to its good chemical and thermal stability [11-14]. Kai-Hsin Chang [15] succeeded in preparing a porous nanofibrous membrane via electrospinning a dilute PSF solution that was dissolved in dimethylacetamide. Xiaoyan Yuan [16] also successfully prepared an electrospun PSF nanofibrous membrane by electrospinning a dilute PSF solution that was dissolved in dimethylacetamide and acetone blending solvents. However, such electrospun PSF nanofibrous membranes are fouled up easily due to their high hydrophobicity and density of electrostatic charges. Therefore, in the present study, oxygen plasma surface modification was used to activate the surface and enhance the surface adsorption energy of the electrospun PSF nanofibrous membrane. After plasma treatment, a low molecular-weight chitosan solution crosslinked by GA was employed to improve its hydrophilicity and mechanical properties. The results obtained in this work are important for the fabrication of electrospun PSF nanofibrous membranes that can be used as water filtration materials.

## **EXPERIMENTAL**

### **Experimental Materials**

PSF resin particles ( $M_w=7.5 \times 10^4$  g/mol) and dimethylformamide (DMF,  $\geq 99\%$ ) were purchased from J&K Chemical Ltd. Chitosan ( $M_w=2 \times 10^4$  g/mol) with approximately 85% deacetylate was purchased from Qingdao Yunzhou Biochemistry Co., Ltd. Acetic acid ( $\geq 99.5\%$ ) and sodium hydroxide ( $\geq 96.0\%$ ) were purchased from Shanghai Chemistry Co., Ltd. Glutaraldehyde (GA,  $\geq 25\%$ )

was purchased from Chinese Medicine Group Chemical Ltd. These were all analytical grade chemicals and were used as received without further purification.

### **Electrospinning**

First, 20wt% PSF spinning solution was prepared by dissolving PSF resin particles in DMF with vigorous magnetic stirring. Then, PSF nanofibrous membranes were prepared by an electrospinning device as follows. First, the prepared solution was fed at a constant rate of 1.5 ml/h into a needle by using a syringe pump with a stainless spinneret (ID 0.35 mm). Then, by applying a 12 kV voltage, electrospinning was performed on aluminum foil located 15 cm from the tip of the needle for 30 min. Second, the electrospun nanofibrous membranes were left in air for 48 h at room temperature to remove the residual solvent in the membranes and then exposed to heat treatment at 190°C for 2 h in an electro-thermoblast oven (DHG-9076A, Shanghai jinghong, Ltd). During the heat treatment, the four sides of the membranes were secured with glass plates to prevent contraction, as shown in *Figure 1*.



FIGURE 1. Scheme of the heat treatment.

### **Oxygen Plasma Pre-treatment**

After heat treatment, the electrospun PSF nanofibrous membranes were treated with oxygen plasma at 50 W and 30 Pa for 3 min at room temperature.

### **Padding Using Chitosan Solution**

Chitosan was dissolved in 0.1 mol/L acetic acid solution to form a 1%-2.5% (w/v) chitosan solution at room temperature. Then, 20-120  $\mu$ l GA solution was mixed with 25 ml chitosan solution using vigorous magnetic stirring for 30 min to form a uniform padding solution. Subsequently, soaked electrospun PSF nanofibrous membranes were pre-treated with oxygen plasma in the padding

solution until complete wetting was obtained. Padding was performed three times at room temperature. After that, the membranes were removed from the padding solution and flattened on the glass plate, and then the excess solution was rolled out until the residual rate of the chitosan solution in the membranes was approximately 80%-100%, as shown in *Figure 2*. Finally, the padded membranes were suspended and dried in an oven at 40°C, and the dried membranes were soaked in 2% sodium hydroxide solution for several hours to remove any residual acetic acid. Double distilled water was used to wash the membranes several times until they were totally neutralized and then dried again at 50°C.

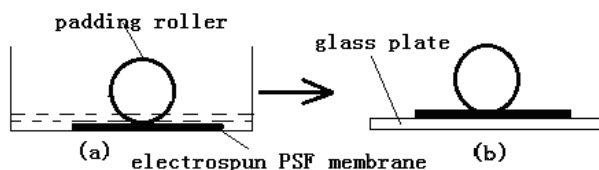


FIGURE 2. Padding process of electrospun PSF membrane, (a) a picture of electrospun PSF membrane padded in chitosan solution, (b) padding excess chitosan solution in membrane.

### **Characterization**

The chitosan was dissolved in 0.1 mol/L acetic acid solution with vigorous magnetic stirring for 1 h at room temperature. Then, GA solution was mixed into it with vigorous magnetic stirring for 30 min. After that, the viscosity of the chitosan solution crosslinked by GA was immediately measured by an NDJ-rotating viscosity meter with a No. 1 rotator and a rotating rate of 60 r/min at 20°C.

A 2 mm×2 mm membrane was fixed on a sample holder with double-sided adhesive tape and coated with gold. Then, longitudinal images of the fibers were observed and recorded using a Hitachi S-4800 SEM. The fiber diameters were obtained with HJ2000 software. First, the number of pixels (P) of the scale line was recorded by the software, and the real length (L) was obtained from the SEM picture. Then, the pixel numbers (p) of the fiber diameter and the real value of the fiber diameter (D) were calculated using the following formula.

$$D(\mu\text{m}) = (L(\mu\text{m}) \times p) / P \quad (1)$$

One hundred fibers were measured for every sample, and the average diameter was calculated.

The porous diameter of the membranes was measured using a Porometer 3G testing meter. First, the 25-mm-diameter circular membrane was soaked in Porofil fluid until it was completely wetted and was then put into the sample channel. Second, the range of testing of the porous diameters was set at 1 μm to 7 μm. Finally, the porous diameter was calculated from the distribution of a wet curve and a dry curve. Each sample was measured three times at different positions.

The surface wettability of a membrane was detected by a water contact angle (WCA) method at room temperature. The water contact angles of different membranes were measured using a Dataphysics OCA contact angle system, and the continuous change of a 2 μl water droplet on the membrane surface was recorded with a camera. The average CA values were obtained by measuring five different positions of the same sample.

The mechanical properties of the membranes were tested using an Instron 3365 mechanical testing machine with a gauge length of 10 mm, testing width of 2 mm, drawing speed of 10 mm/min, initial tension of 0.2 cN, strength of 0.01 cN, elongation of 0.01 mm, temperature of 20±2°C and relative humidity of 65±5%. Each sample was balanced for 24 h and tested 20 times.

The thickness of each membrane was measured by an electronic digital micrometer (Guilin Guanglu Measuring Instrument CO., LTD.). Each membrane was measured five times, and the average value was recorded as the final thickness.

The mechanical properties were characterized based on the following formula:

$$\text{Breaking stress (MPa)} = \frac{\text{breaking strength (N)}}{\text{thickness (mm)} \times \text{width (mm)}}$$

$$\text{Breaking strain (\%)} = \frac{\text{breaking length} - \text{sample length (mm)}}{\text{sample length (mm)}} \times 100\%$$

## **RESULTS AND DISCUSSION**

### **Viscosity of Chitosan Solution**

The viscosity of the chitosan solution is related to the amount of chitosan and GA. The reactive mechanism between GA and chitosan is shown in *Figure 3*, which is based on the Schiff Base Imine Functionality or Michael-type Adducts with

Terminal Aldehydes. As shown in *Figure 3*, amine groups of the chitosan macromolecules reacted with aldehyde groups of the GA molecules to form stable reticular cross-linked structures between the chitosan macromolecules. As shown in *Figure 4*, over the range of all of the concentrations that were tested, the viscosity increased with the increase in chitosan concentration and GA volume, but the chitosan solution attained a gel state immediately after mixing 100  $\mu\text{l}$  GA in 2.5% chitosan solution. It is necessary to point out that the rate of increase of the viscosity is directly related to the GA volume, which is slow when the GA volume is below 80  $\mu\text{l}$ , and fast above 100  $\mu\text{l}$ . These results suggest that there is a direct, positive link between the volume of GA and the viscosity due to the reaction between the amine groups of the chitosan macromolecules and the GA.

#### **Effects of Chitosan on the Morphology and Porous Structure of Electrospun PSF Nanofibrous Membranes**

The viscosity of the cross-linked chitosan solution and the residual amount of chitosan after padding has a great influence on the morphology and porous structure of the electrospun PSF nanofibrous membrane. The residual amount of chitosan can be controlled by varying the padding pressure and padding times. Usually, this is expressed in terms of the weight gain rate of the membrane.

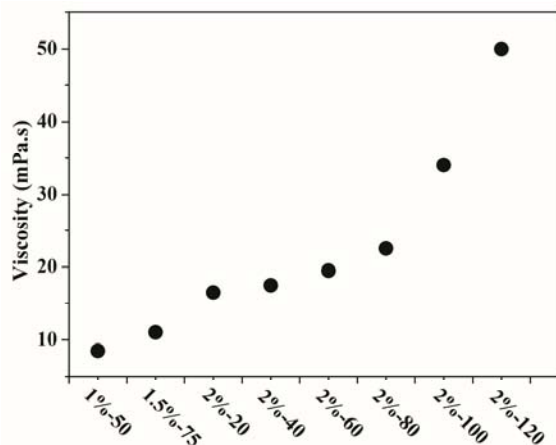


FIGURE 4. Viscosity of chitosan solution crosslinked by GA.

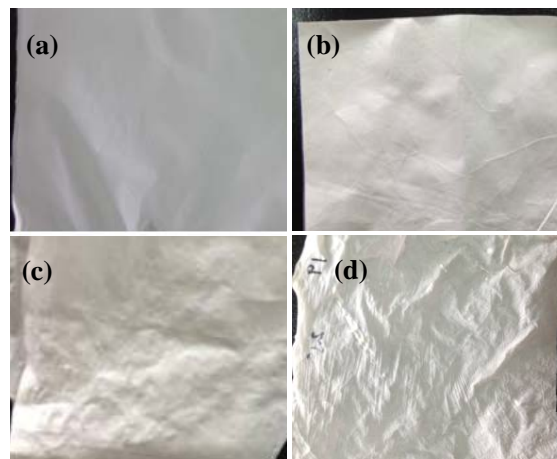


FIGURE 5. Morphology images of PSF electrospun nanofiber membranes before and after padding with different chitosan solutions crosslinked by GA for untreated (a), 2%-120(c), and 2.5%-100(d). The morphologies of the membranes after padding with 1%-50, 1.5%-75, 2%-20, 2%-40, 2%-60, 2%-80 and 2%-100 were similar, and so they are all represented by (b).

*Figure 5* presents the morphology images of the electrospun PSF nanofibrous membranes before and after padding with crosslinking chitosan solution, and *Figure 6* presents their SEM images. As shown in *Figure 5 (a)*, the surface of the untreated electrospun PSF nanofibrous membrane was smooth and dense, and the average fiber diameter was  $663 \pm 254$  nm. As shown in *Figure 5 (b)*, the surface morphology of the membranes showed no significant change after padding with 1%-50, 1.5%-75, 2%-20, 2%-40, 2%-60, 2%-80 and 2%-100 chitosan solution. However, these membranes all absorbed a little chitosan, which was distributed unevenly, as shown in *Figure 6 (b ~h)*, and the pores between the fibers of these membranes were not completely blocked. After padding with 2% - 120 chitosan solution, the surface of the membrane became rough and uneven as shown in *Figure 5 (c)*, and parts of the pores were completely blocked as shown in *Figure 6 (i)*. In contrast, the surface of the membrane became more rough and uneven after padding with 2.5%-100 as shown in *Figure 5 (d)*, and the pores became filled with lots of chitosan particles as shown in *Figure 6 (j,k)*.

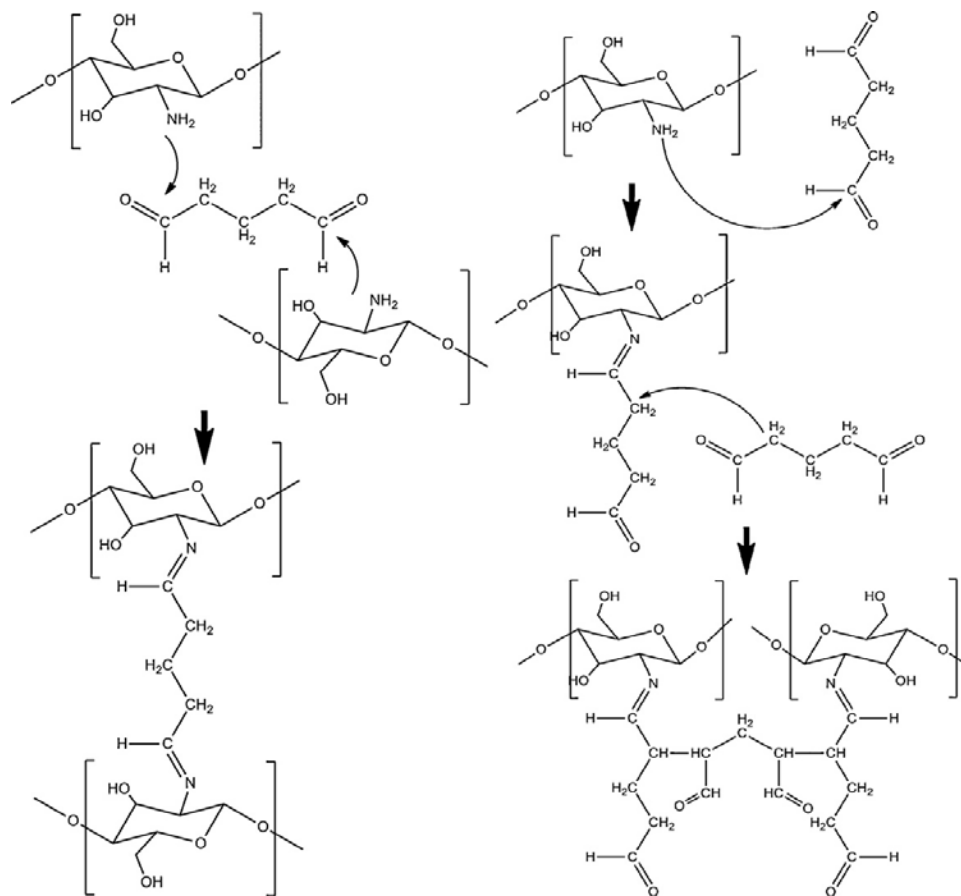


FIGURE 3. Mechanism of chitosan reacting with GA.

TABLE I. Characteristics of electrospun PSF nanofiber membrane before and after padding with different chitosan solutions cross-linked by GA.

Chitosan concentration (%)	GA volume ( $\mu$ l)	Thickness of membrane before padding ( $\mu$ m)	Thickness of membrane after padding ( $\mu$ m)	Padding residual rate (%)	Residual amount of chitosan (%)
untreated	--	54	54	--	--
1.0	50	54	52	98.5	11.1
1.5	75	51	49	96.9	15.3
2	20	53	54	80.5	13.0
	40	55	56	81.4	13.2
	60	53	52	82.5	13.4
	80	54	51	88.1	14.7
	100	50	45	90.8	14.9
	120	49	33	97.8	20.4



The viscosity of the chitosan solution has a great influence on the evenness and residual amount of chitosan in the membranes when the padding pressure and padding times are certain. *Table I* show that the residual amount of chitosan was smaller after padding with 1%-50, owing to its lower viscosity of approximately 8.5 mPa . When the padding solution was 2%-120, it was easy to form the film and block the pores between the fibers due to its larger viscosity (approximately 50 mPa.) and increased residual amount of chitosan. However, other membranes almost have no change after padding with 1.5%-75, 2%-20, 2%-40, 2%-60, 2%-80 and 2%-100. Therefore, the viscosity and the residual amount of chitosan should be strictly controlled so that the fibers can adsorb some chitosan but do not block the pores. On the other hand, the viscosity of the chitosan has a great effect on the thickness of the membrane. The thickness of the membrane almost has no change after padding

with 1%-50, 1.5%-75, 2%-20, 2%-40, 2%-60 and 2%-80 because these solutions have lower viscosity and the residual amount of chitosan is smaller. The thickness of the membrane decreased by 10% and 32.7% after padding with 2%-100 and 2%-120, respectively, because the fiber layers had been pressed to each other and bonded together. In addition, the surface of the membranes became rougher and more uneven due to the larger viscosity, which made the chitosan solution hard to flow. Therefore, the solution could not permeate into the membranes and the chitosan was distributed unevenly within the membranes. When the chitosan solution was increased to 2.5%-100, it attained a gel state and it was unable to permeate into the membrane and most of the solution thus stayed on the surface. At such a time, if we were to apply horizontal and vertical forces to the gel at the same time, the gel would become small particles.

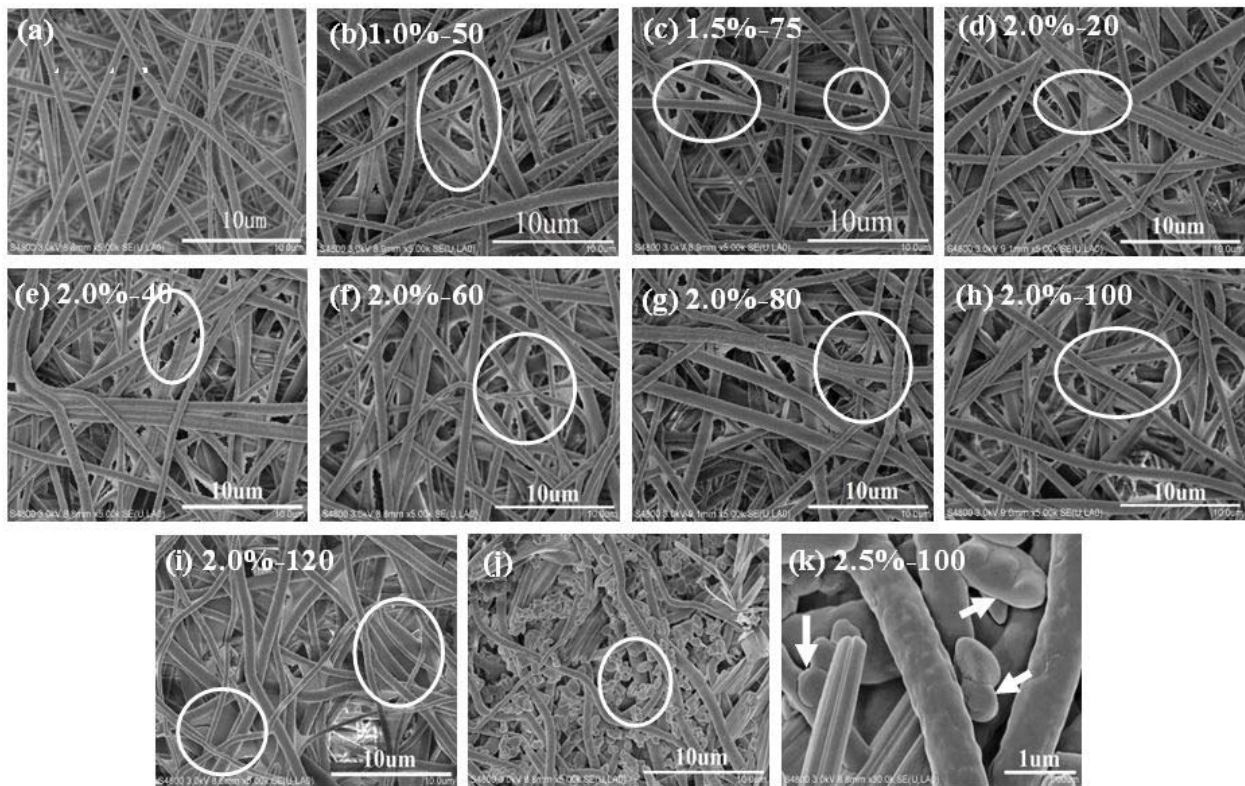


FIGURE 6. SEM images of PSF electrospun nanofibrous membranes before and after padding with different chitosan solutions crosslinked by GA.

As shown in *Figure 7*, the pore sizes of the membranes decreased significantly after padding with chitosan solution cross-linked by GA. The average pore size of the untreated membrane was 4.5  $\mu\text{m}$ . However, this pore size decreased gradually and the range between the maximum and minimum pore size decreased as the chitosan concentration (%) was increased when the ratio of the GA volume ( $\mu\text{l}$ ) to chitosan concentration was 50 to 1. Meanwhile, when the chitosan concentration was 2%, the average pore size of the membranes decreased gradually with increasing GA volume from 20  $\mu\text{l}$  to 100  $\mu\text{l}$ . When the padding solution was 1.5%-75, the average pore size of the membrane was 2.75  $\mu\text{m}$ , which was smaller than 3.1  $\mu\text{m}$  and 3.05  $\mu\text{m}$  after padding with 1%-50 and 2%-20 chitosan solution, respectively. Because the residual amount of chitosan was larger after padding with 1.5%-75, the fibers adsorbed more chitosan, which made the pore size decrease. When the padding solution was 2%-100, the average pore size of the membrane decreased to 2.68  $\mu\text{m}$  due to its large viscosity. Thus, it can be seen that the pore size of the membranes was consistent with the viscosity of the chitosan solution and the residual amount of chitosan. However, the pore size of the membrane decreased significantly and the pores were covered by chitosan film after padding with 2%-120, and so the pore size could not be measured accurately.

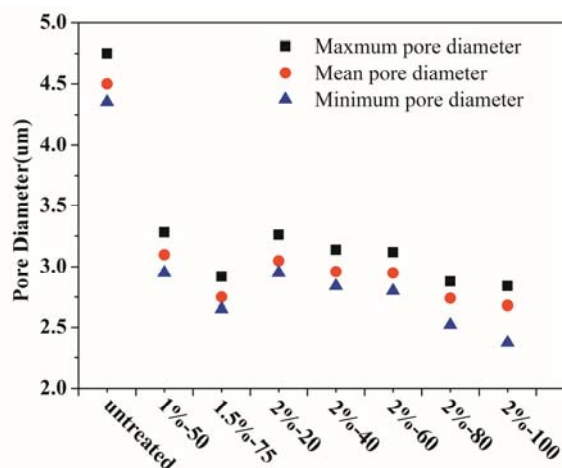


FIGURE 7. Pore diameters of electrospun PSF nanofiber membranes padded with different chitosan solutions crosslinked by GA.

### Effects of Chitosan on the Mechanical Properties of Electrospun PSF Nanofibrous Membranes

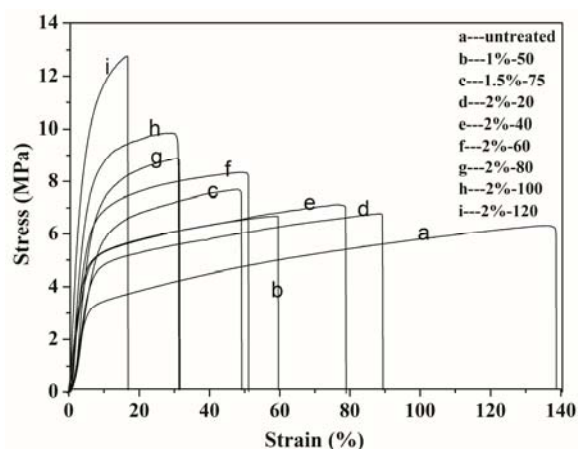


FIGURE 8. Tensile curves of electrospun PSF nanofiber membranes before and after padding with GA cross-linked chitosan solution.

As observed from *Figure 8*, the tensile curves of the electrospun PSF nanofibrous membranes are typical stress-strain curves of viscoelastic materials that have obvious yield points and smooth before and after padding with cross-linked chitosan solution. As shown in *Figure 9* and *Figure 10*, the breaking stress and Young's modulus increased gradually, but the breaking strain decreased significantly with increasing chitosan concentration when the ratio of GA volume to chitosan concentration was 50 to 1. Compared to the untreated membrane, the breaking stress and Young's modulus of the membrane padded with 1%-50 chitosan solution showed no obvious change, but the breaking strain decreased by 58.8% from  $139.38 \pm 9.25\%$  to  $57.49 \pm 13.28\%$ . After padding with 2%-100, the breaking stress increased by 53.9% from  $6.01 \pm 0.44$  Mpa to  $9.25 \pm 0.45$  Mpa, and Young's modulus increased by 134.7% from  $75.58 \pm 4.1$  Mpa to  $177.42 \pm 10.81$  Mpa, but the breaking strain decreased by 77.2% from  $139.38 \pm 9.25\%$  to  $31.78 \pm 4.36\%$ .

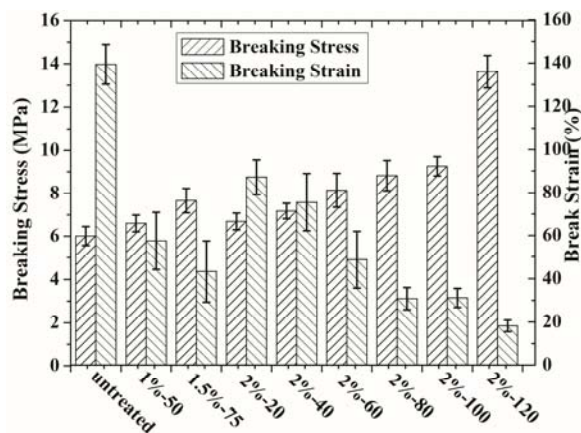


FIGURE 9. Mechanical properties of electrospun PSF nanofiber membranes before and after padding with GA cross-linked chitosan solution.

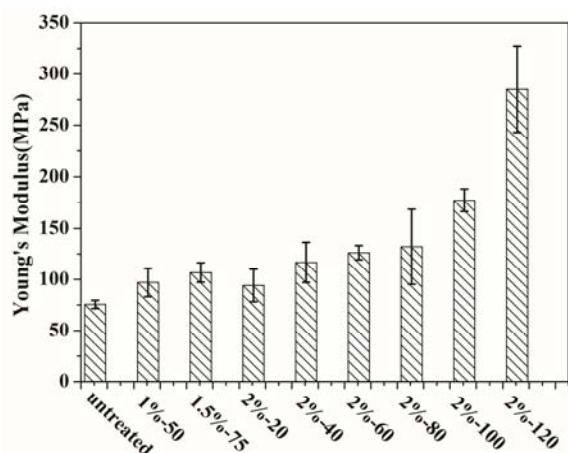


FIGURE 10. Young's modulus of electrospun PSF nanofiber membranes before and after padding with GA cross-linked chitosan solution.

When the chitosan concentration was 2%, the breaking stress and Young's modulus of the membranes increased gradually, and the breaking strain decreased gradually as GA volume was increased from 20  $\mu$ l to 120  $\mu$ l. Compared to the untreated membrane, the breaking stress and Young's modulus of the membrane changed less after padding with 2%-20, but the breaking strain

decreased by 37.5%. The breaking stress and Young's modulus showed no obvious change between 2%-60 and 2%-80, but the former increased by 35.4% and 46.8%, respectively, and the latter increased by 67.0% and 74.8%, respectively, compared to untreated membrane. However, the breaking strain decreased by 65.0% and 72.2%, respectively. When the padding solution was 2%-120, the breaking stress and Young's modulus increased by 126.8% and 277.2%, respectively, compared to the untreated membrane, but the breaking strain decreased by 86.9%.

The fiber structure, geometry and bonding force between the fibers all have great influence on the mechanical properties of the electrospun PSF nanofibrous membranes. In general, the electrospun nanofibrous membrane has a lower tensile stress due to its sub-micrometer diameter, low orientation and crystallinity. In addition, the bonding force between the fibers is low, and the fibers can thus easily slip along the tensile direction, resulting in lower breaking stress and larger breaking strain. In this study, the fibers in the membrane were bonded together after padding with cross-linked chitosan solution, and the slipping between the fibers therefore decreased, which resulted in a larger breaking stress and a lower breaking strain. The concentration and viscosity of the chitosan solution greatly influence the binding force between the fibers. When the concentration was 2%, the viscosity of the chitosan solution increased with the GA volume so that a larger binding force was produced between the fibers. This resulted in a larger breaking stress and Young's modulus and a lower breaking strain due to the reduced slippage between the fibers during elongation. As mentioned above, the same result occurred with increasing chitosan concentration when the ratio of chitosan concentration to GA volume was 50 to 1.



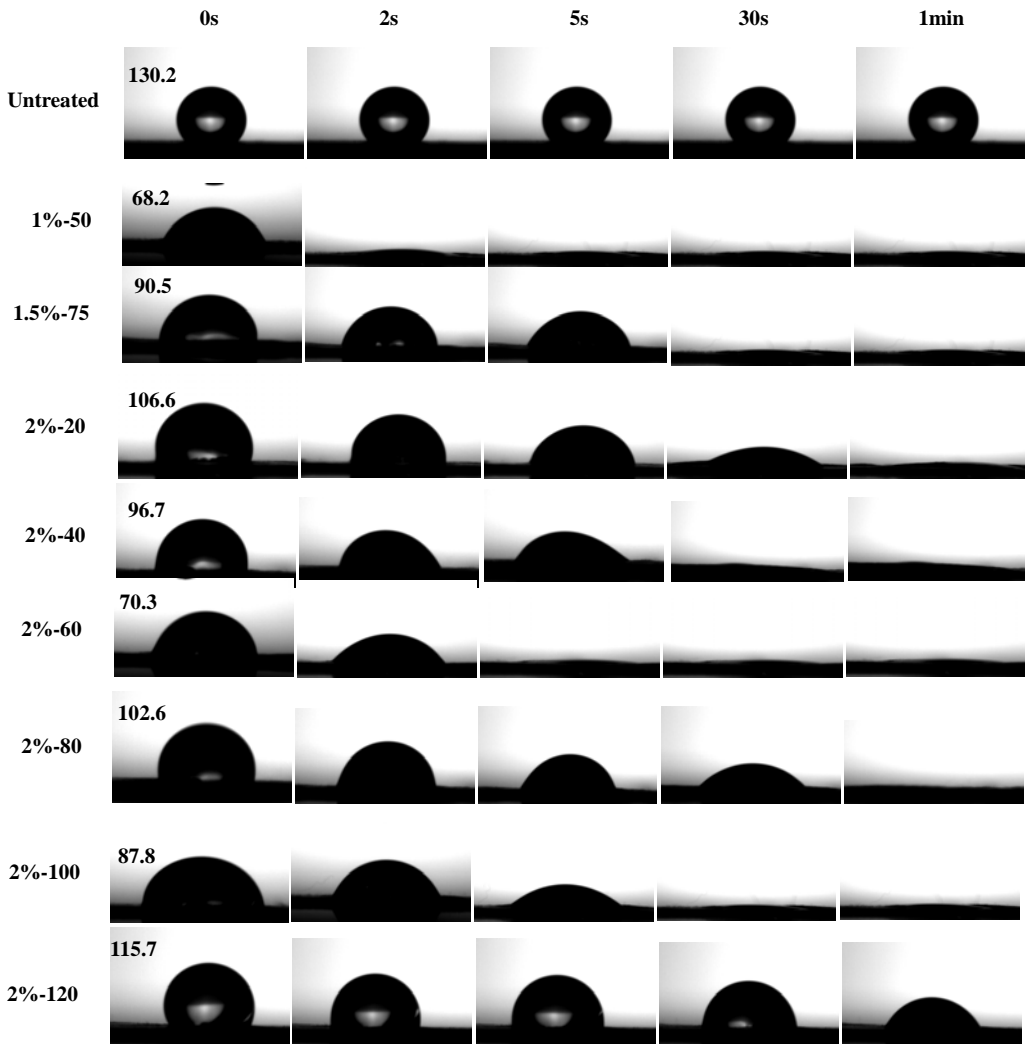


FIGURE 11. Images of water sessile drop on all samples.

### **Effects of Chitosan on the Surface Wettability of Electrospun PSF Nanofibrous Membranes**

The dynamic water-drop deformation is shown in *Figure 11* as a function of time. PSF is a hydrophobic polymer, and the electrospun fibers have relatively high roughnesses [17]; thus, the water contact angle on the surface of the as-electrospun PSF nanofibrous membrane was up to 130.2°. As we know, to change the hydrophilicity of the highly rough surface, the surface energy of the liquid/solid interface is a dominant factor. In our study, the chitosan provided the hydrophilic component, and pores in the membranes result in wicking. Therefore, the hydrophilicity of the electrospun PSF nanofibrous membrane was significantly improved after padding with chitosan

cross-linked by GA. As shown in *Figure 11*, after being padded with chitosan solution cross-linked by GA, a 2  $\mu$ l water drop deformed quickly, spread out on the surface, and penetrated into the membranes within 1 min of observation (except in the 2%-120 case). However, there was no obvious regularity in the spreading speed of the water drops on the electrospun PSF membranes. This result may indicate that the cross-linked chitosan is distributed unevenly in the membranes and that the GA was unevenly distributed in the chitosan solution. When the membranes were padded with the 2%-120 solution, the bulk of the pores in the membrane were blocked by cross-linked chitosan, as shown in *Figure 6 (i)*. As a result, wicking decreased greatly, and more GA reacted with the chitosan so that the

hydrophilic groups of the chitosan were greatly reduced. Therefore, the spreading speed of the membrane padded with 2%-120 was the slowest.

## CONCLUSIONS

In this study, oxygen plasma was used to pre-treat electrospun PSF nanofibrous membranes. The membranes were then padded with different GA cross-linked chitosan solutions to improve the mechanical properties and hydrophilicity of the electrospun PSF nanofibrous membrane. The results showed that the electrospun PSF membranes adsorbed a little chitosan, and the mechanical properties and hydrophilicity were improved noticeably. Pores were still present, but they became smaller. The optimum concentration was 2%-100. In this condition, the average pore size was 2.68  $\mu\text{m}$ , the breaking stress was 9.25 $\pm$ 0.45 Mpa (having increased by 53.9% compared with untreated membrane), and the water drop could spread and permeate into the membrane within 30 s. Therefore, these electrospun PSF nanofibrous membranes (after padding with chitosan) have huge potential for application in water filtration systems.

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