Preparation of poly (lactic-co-glycolic acid) microfiber membrane

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Abstract. Poly (lactic-co-glycolic acid) (PLGA) is a kind of medical polymer material with good biodegradability and histocompatibility, which is widely used in postoperative anti-adhesion materials. In this paper, PLGA microfiber membrane was successfully prepared with poly(lactic-co-glycolic acid) as the main material by electrospinning technology. The surface morphology, wettability, dimensional stability and mechanical properties of PLGA microfiber films were characterized by scanning electron microscopy (SEM) and tensile testing machine. The results show that the average diameter of the prepared PLGA fiber is 2.218~2.313 µm, the elongation at break of the fiber film is up to 193%, and the dimensional stability and wettability of the fiber film are maintained at 37°C. The degradation cycle is 2~3 months.

Keywords: Electrospinning, poly(lactic-co-glycolic acid), fiber diameter, adhesion

1. Introduction

Poly(lactic-co-glycolic acid) is an environmentally friendly biopolymer widely used in anti-adhesion due to its excellent biodegradability and biocompatibility as well as good mechanical properties^[1-3]. The PLGA isolation membranes that are currently popular in the market are mostly formed by casting method. This preparation method has the characteristics of smooth and easy sliding surface of the isolation membrane. Generally, it needs to be sutured and fixed during the implantation process, which increases the probability of adhesion.

The non-woven fabric prepared by electrospinning technology has the advantages of high porosity, large specific surface area, high fiber fineness and uniformity, and large length-to-diameter ratio^[4-5]. It is several orders of magnitude smaller than the fiber diameter prepared by conventional methods. These advantages It has many practical and potential application values.

In this paper, a kind of PLGA microfiber membrane with good operability and good structural compatibility was prepared by electrospinning technology with polyvinyl lactide as the main raw material, which can avoid strong discomfort after implantation of anti-adhesion fiber membrane in patients.

2. Experiment

2.1 Main materials

The intrinsic viscosity ($[\eta]$) of polylactide (PLGA) was 0.5~0.7. Sodium dihydrogen phosphate, dipotassium hydrogen phosphate, N, N-dimethylformamide (DMF)

and acetone are all analytically pure (AR), which are provided by Aladdin Chemical Reagent Co., Ltd., etc.

2.2 Main instruments

Vacuum drying oven (Shanghai Senxin experimental instrument). Electrospinning apparatus (E05, Lepton apparatus). Microcomputer control electronic universal testing machine (Shenzhen Wance Testing Machine Co., Ltd). Scanning electron microscope (Inspect-S50, FEI, USA). Ubbelohde viscometer, rotary viscometer, etc.

2.3 Preparation of PLGA Microfiber membrane

Add PLGA, DMF, and acetone in a certain proportion into a sealed reagent bottle with magnetic stirring, and stir until completely dissolved. Use electrospinning instrument to prepare PLGA microfiber membrane, and place it in a 25°C vacuum drying oven for 24 h to remove the residual organic solvent in the fiber membrane. Then, the performance of the fiber membrane was measured, and the voltage, solution concentration, injection speed and other parameters were adjusted according to the test results. Finally, the smooth and uniform surface of the PLGA microfiber membrane was obtained.

2.4 Performance characterization

2.4.1 Dimensional stability

Cut the PLGA fiber membrane into 20 mm×20 mm samples and place them in PPS at 37°C for 15 minutes. Take out and measure the sample size and calculate its

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retention area, which is the proportion of the initial sample area.

2.4.2 Wettability

Cut the PLGA fiber membrane into 20mm×20mm samples and place them in PPS at 37°C. Observe the time required for the fiber membrane to fully wet.

2.4.3 Determination of elongation at break

According to the international standard ISO1184, the mechanical properties of PLGA fiber membrane are measured.

2.4.4 Microtopography measurement

The micro morphology of polyglycolide Microfiber membrane was observed with the scanning electron microscope.

3. Results and Discussion

3.1 Influence of solution concentration on PLGA microfiber membrane

The viscosity of the spinning solution directly affects the morphology and properties of the microfibers produced by electrospinning. The larger the viscosity of the spinning solution, the more easily the polymer molecular chain entanglement, the more unstable the jet, the more difficult the spinning is, and it is difficult to produce microfibers with uniform diameter distribution. However, the low viscosity cannot form a jet, only droplets can be formed, so preparing a spinning solution with a suitable viscosity is the key first step in electrospinning. In this paper, the effects of different solution concentrations on the solution viscosity and the properties of PLGA fiber membranes are studied, as shown in Figure 1 and Table 1.

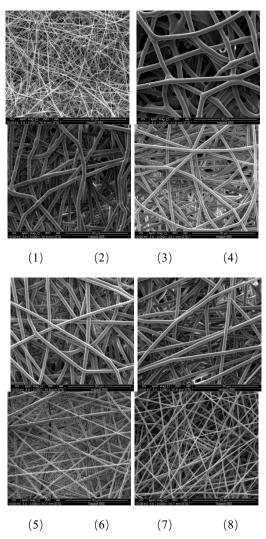


Figure 1. The effect of solution viscosity on the morphology of PLGA fiber membrane.

Number	Concentration /(g/mL)	Viscosity /(mPa·s)	Dimensional stability/%	Fibre diameter/nm	Appearance
1	0.55	6120	69.59		rough
2	0.5	4210	78.90	2.535	smooth
3	0.475	3650	81.75	2.308	smooth
4	0.45	2970	74.39	2.235	smooth
5	0.425	2200	65.61	2.143	smooth
6	0.4	1670	52.56	1.773	smooth
7	0.375	1540	44.30	1.342	smooth
8	0.35	1100	34.55	1.0902	fluffy

Table 1. Effect of solution viscosity on properties of PLGA fiber membrane

As shown in Figure 1 and Table 1, with the increase of solution concentration, from 0.35 g/mL to 0.55 g/mL, the solution viscosity ranges from 1100 to 6120 mPa·s. When the solution viscosity ranges from 1670 to 3650 mPa·s, the prepared fiber film has a smooth and uniform appearance. Under certain other conditions, when the solution viscosity is greater than 3650 mPa·s, the electric field force cannot overcome the surface tension and viscous force of the solution, the fibers are thicker, the solvent cannot be fully volatilized, the fibers appear to be cross-linked, and the viscosity continues to increase to 6120 mPa·s, the fiber filaments cannot be stretched evenly, and the phenomenon of beading appears. When the viscosity is too low, the electric field force is much greater than the surface tension and viscous force of the solution, the fiber filaments are stretched excessively, broken filaments appear, the fiber membrane is fluffy and the strength is low.

3.2 Effect of voltage on PLGA microfiber membrane

Under the same conditions of other process parameters, the effect of applied voltage on the morphology of PLGA fiber film was studied, and the results are shown in Figure 2 and Table 2.

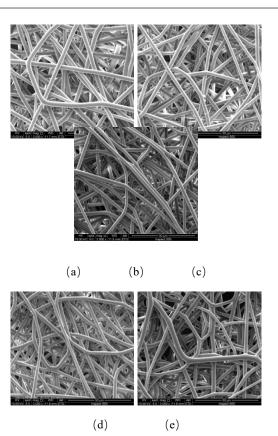


Figure 2. Effect of applied voltage on the morphology of PLGA fiber membrane.

Number	Voltage /kV	Fibre diameter /nm	Fiber film rollout	Appearance
а	12	2.511	difficult	rough surface
b	16	2.341	less difficult	rough surface
с	20	2.235	easy	uniform and smooth
d	24	2.218	easy	uniform and smooth
e	28	2.821	difficult	rough surface

Table 2. Effect of applied voltage on properties of PLGA fiber membrane

As shown in Figure 2 and Table 2, when the applied voltage is 12~24 kV, as the voltage increases, the fiber diameter gradually becomes smaller, the fiber thickness uniformity changes from poor to better, and the graininess on the surface of the spinning membrane gradually decreases to become Smooth, the diameter of the fiber is almost unchanged when the voltage is 20 kV and 24 kV, as the voltage continues to increase at 28 kV, the jet is directly ejected from the needle tube, the formation of the Taylor cone is unstable, and tiny particles appear on the surface of the spinning film, and appear thicker fibers. When the voltage is greater than 28 kV, the jet is ejected directly from the needle tube, the Taylor cone cannot be formed, and the phenomenon of floating filaments occurs, which is not conducive to the collection plate and cannot form a fiber film.

3.3 Effect of injection speed on PLGA microfiber membrane

Under the condition that other processes remain unchanged, the influence of injection speed on the performance of PLGA fiber membrane is studied, and the results are shown in Figure 3 and Table 3.

(H)

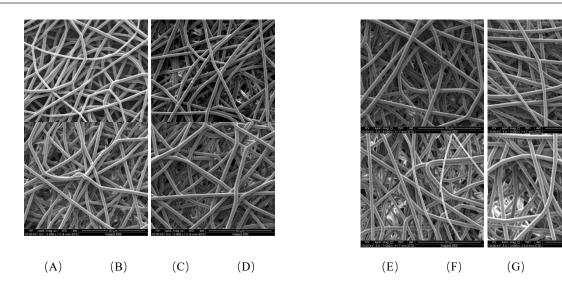


Figure 3. The effect of injection speed on the morphology of PLGA fiber membrane.

Number	Speed /(mL/h)	Fibre diameter/µm	Fiber film rollout	Appearance
А	0.4	1.780	difficult	rough surface
В	0.6	2.160	less difficult	rough surface
С	0.8	2.201	less difficult	uniform and smooth
D	1.0	2.218	easy	uniform and smooth
E	1.2	2.223	easy	uniform and smooth
F	1.4	2.313	easy	uniform and smooth
G	1.6	2.452	less difficult	rough surface
Н	1.8	2.845	less difficult	rough surface

Table 3. The effect of injection speed on the properties of PLGA fiber membrane

As shown in Figure 3 and Table 3, when the injection speed is from 0.4 to 0.8 mL/h, when the injection speed is small, due to the inability to provide enough precursor solution, the size of the jet ejected from the Taylor cone is unstable, and splitting occurs. The double-column jet flow is formed, the fiber thickness is uneven, the fiber continuity is poor, and the surface of the fiber film is rough, as the injection speed increases, at 1.0~1.4 mL/h, the jet gradually stabilizes, and the fiber diameter decreases and is relatively uniform. The fiber diameter ranges from 2.218 to 2.313 µm, when the injection speed continues to increase, that is, 1.6 to 1.8 mL/h, the amount of solution injected per unit time increases, causing the fibers to fail to completely volatilize during the flight, forming a bonded Fiber, the degree of dispersion of fiber diameter is reduced, and the distribution of fiber diameter is larger. Therefore, the best injection speed is $1.0 \sim 1.4$ mL/h, preferably 1.2 mL/h.

3.4 Performance determination of PLGA microfiber membrane

Using electrospinning technology, the preparation process parameters are as follows: the concentration of spinning precursor solution is 0.45 g/mL, the voltage is 20 kV, the injection speed is 1.2 mL/h, the PLGA ultrafine fiber membrane is prepared and measured, and the results are shown in the Table 4 shown.

Table 4. Properties of PLGA microfiber membrane

Project	Consequence	
Film thickness/µm	110	
Dimensional stability /%	74	
Wetting time /s	55	
Elongation at break/%	193	
Degradation cycle/Mth	2~3	

As shown in Table 4, under the PPS environment at 37° C, the PLGA microfiber membrane retains an area of 74%, the wetting time is only 55 s, and the degradation cycle is 2-3 months. The fibrous membrane exhibits good dimensional stability and hydrophilicity, as well as a suitable degradation rate.

4. Conclusion

According to the experimental results, the optimum spinning process parameters were determined: the concentration of spinning precursor solution was 0.45 g/mL, the voltage was 20 kV, the injection distance was 16cm, the injection speed was 1.2mL/h, and the injection time was 220 min. The average diameter of the PLGA fiber obtained by using this parameter is $2.218 \sim 2.313$ µm, the elongation at break of the fiber film is as high as 193%, and the degradation time is $2 \sim 3$ months. It also has good dimensional stability and hydrophilicity, and can be used in Postoperative anti-adhesion material.

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