Maneuvering the secondary surface morphology of electrospun poly (vinylidene fluoride) nanofibers by controlling the processing parameters

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Abstract
Tailoring surface of fibers has been attracting the attention of researchers in different fields and applications. Nowadays, appreciations to the electrospinning technique, polymeric nanofibers are easily producible. The electrospinning process has been prominently investigated and developed during the last decade. The influence of working parameters on the secondary surface morphology of electrospun fibers is very significant. In this study, the effect of processing parameters (applied voltage, flow rate, distance between the tip of the needle and the collector (DTC), diameter of the needle, and rotation speed of the drum collector) on the secondary surface morphology (e.g. porous, grooved, and rough) of electrospun poly (vinylidene fluoride) (PVDF) fibers are studied. The results indicate that the secondary surface morphology of electrospun PVDF fibers can be alerted by maneuvering applied voltage, flow rate, DTC, and rotation speed of the drum collector. However, there is no relationship between the secondary surface morphology of electrospun PVDF fibers and the diameter of the needle. Importantly, fibers with different secondary surface morphologies have the ability to be served in different applications such as energy harvesting, oil cleanup, filtration, and so on. We believe this study can be served as a good reference for generating electrospun fibers with the desired structure by controlling the processing parameters.

1. Introduction

Since the beginning of human existence on Earth, they have advanced command of subtle structural characteristics that are significant to their continued existence.

Secondary surface morphology of materials obtained at the nanoscale is considered one of the most important factors affect their properties and applications. In recent years, a considerable number of researches have focused on the secondary surface morphology of fibers at the nanometer scale to understand deeply their advantages - related properties [1–4].

Electrospinning is a technique for generating fibers by pumping and elongating polymer jets based on electrostatic forces [5]. The diameter of fibers obtained from this technique could be ranged from nanometers scale to micrometers scale [6–10]. Different morphologies of fibers can be produced via electrospinning such as grooved fibers [11], wrinkled fibers [12], porous fibers [13], rough fibers [3], rice grain-shaped nanocomposites [14], core-sheath fibers [15], hollow fibers [16], crimped fibers [17], ribbon fibers [18], butterfly wings fibers [19], side-by-side fibers [20], cactus-like fibers [2], tree-like fibers [21], round fibers [22], and so on. Since electrospun fibers have exceptional properties (e.g. ease of functionality [23], small diameters [24], good pore...
structures [5], high porosity [23], low density [25], excellent mechanical properties [26], flexibility [5], and a variety of morphologies and structures [27]), they can be used in multiple applications such as catalyst [28], food packaging [29], filtration [30, 31], superhydrophobic surfaces [32, 33], sensors [34, 35], biomedical applications [36, 37], energy harvesting [1, 38–40], and so on.

Studies have proved that the secondary surface morphology of electrospun fibers can strongly affect their behavior and properties. For example, cactus-like nanofibers have shown great potential in the field of self-cleaning surfaces as well as energy scavenging [2]. Porous fibers have displayed wide use in different areas such as oil cleanup, catalysis, biomedical research, and filtration [3, 41]. Grooved fibers have been served successfully in self-cleaning surfaces and tissue engineering applications [42]. Rough fibers have been used to increase the electrical output of the energy harvesting devices [43].

Numerous parameters in the electrospinning procedure comprising the solution parameters (polymer concentration, viscosity, molecular weight, surface tension, and conductivity), processing parameters (applied voltage, flow rate, collector, distance between the tip of the needle and the collector (DTC), and diameter of the needle), and ambient parameters (relative humidity and temperature) can affect the morphologies and properties of fibers obtained [12, 44–51].

Previously, we have investigated the effect of the solution parameters on the secondary surface morphology of electrospun poly(vinylidene fluoride) (PVDF) fibers [12, 27]. Furthermore, our group has explored the relationship between the ambient parameters and the secondary surface morphology of PVDF fibers and their possible applications [2, 3].

In this study, we demonstrate the effect of the processing parameters on the secondary surface morphology of PVDF fibers fabricated via electrospinning without involving any post-spinning treatment or special collection method. Herein, PVDF was selected as a model because it can be used in various applications owing to its unique properties such as piezo, pyro- and ferroelectricity, high sensitivity, good flexibility, good chemical resistance, high thermal stability, membrane forming properties, its ability to be dissolved in many solvents, and formed in different structures [27, 39, 52–54].

To the best of our knowledge, up to now, no studies have been systematically investigated the effect of processing parameters on the secondary surface morphology of PVDF fibers. We determined the relationship between the processing parameters and the secondary surface morphology of electrospun PVDF nanofibers. We concluded that the processing parameters play a significant role in defining the secondary surface morphology of PVDF fibers. This study can be used as a good reference for maneuvering the surface structure of electrospun fibers by tailoring the processing parameters.

2. Experimental

2.1. Materials
PVDF pellets at a molecular weight of 275000 were purchased from Sigma-Aldrich, USA. Acetone (ACE) and N, N-dimethylformamide (DMF) were purchased from Shanghai Chemical Reagents Co., Ltd, China.

2.2. Methods
Electrospinning: 14% (w/v) PVDF solution with ACE, 22% (w/v) PVDF solution with DMF, and 27% (w/v) PVDF solution with ACE/DMF at the solvent ratio of 4:1 were prepared and loaded in the plastic syringe. The polymer concentrations and the solvents used were selected based on their effects on the secondary surface morphology and the spinnability of the fibers [27]. Herein, the solvent ratio was the volume ratio, and the solution concentration was the weight/volume (w/v) (g/ml). A syringe needle was used as the spinneret, which was fixed on a syringe pump (KDS 100, KD Scientific Inc., USA) connected to a high-voltage supplier (Tianjin Dongwen Co., Ltd, China). A grounded drum collector (40 cm in length and 20 cm in diameter) was used to obtain randomly-oriented fibers and aligned fibers by controlling its speed. All the experiments were carried out with an applied voltage of 6–24 kV; flow rate of 1–2.5 ml h⁻¹, distance between the tip of the needle and the collector (DTC) of 8–24 cm, needle diameter of 0.1–0.83 mm, and rotating collector speed of 2–3000 rpm (figure 1). All of the samples were prepared at the relative humidity of 65% and temperature of 20 °C [3].

2.3. Characterization
The surface morphology of the electrospun PVDF fibers was detected under field emission scanning electron microscopy (FE-SEM, S-4800 Hitachi, Japan). Fiber diameter was detected using image processing software (Image) 1.45s.
3. Results and discussion

To explore the effect of the processing parameters on the secondary morphology of electrospun PVDF fibers, 14% (w/v) PVDF solution with ACE, 22% (w/v) PVDF solution with DMF, and 27% (w/v) PVDF solution with (ACE/DMF: 4/1) were electrospun to get porous, rough, and grooved structures, respectively, at five independent variables including applied voltage, flow rate, distance between the tip of the needle and the collector (DTC), diameter of the needle, and rotating speed of the collector.

3.1. Effect of the applied voltage on the secondary surface morphology of PVDF fibers

In order to determine the relationship between the applied voltage and the secondary surface morphology of PVDF fibers, PVDF fibers were electrospun at different applied voltages while other processing parameters were kept constant as shown in table 1.

For porous fibers, the shape of macroporous on the surface of fibers altered to elliptical by increasing the applied voltage from 6 kV to 24 kV (figures 2(a)–(d)). However, the dimension of pores formed was decreased at an applied voltage of 24 kV. When the highly volatile solvent (ACE) evaporated, it absorbed a huge amount of heat and cooled the surface of the fiber resulting in condensing and attracting water droplets on the surface of fibers (Thermal induced phase separation) [3]. Afterward, the combination between the droplets formed macro droplets because of the nucleation growth (NG) mechanism [55]. By increasing the applied voltage from 6 kV to 18 kV, the repulsive forces stretched the jet resulting in forming elliptical pores. However, above 18 kV the repulsive forces will be too high because of the increase in the electric field. Therefore, that will not give enough time to water droplets to combine with each other resulting in forming smaller elliptical pores [6].

Regarding grooved fibers, the number of grooves on the surface of fibers increased by increasing the applied voltage from 6 kV to 18 kV, but they decreased at the applied voltage of 24 kV (figures 2(e)–(h)). Herein, due to the presence of DMF (low volatile solvent), the porous which were formed via thermal-induced phase separation were elongated into the grooved structure. The increase in the number of grooves by raising the applied voltage up to 18 kV should be attributed to the positive relationship between the applied voltage and the electric field [48]. When the applied voltage increases, the electric field will increase resulting in not giving the pores enough time to combine with each other. In other words, when the number of pores increases, the number of grooves will increase.

For rough fibers, the roughness degree of the fibers surface increased by raising the applied voltage from 6 kV to 18 kV, but it decreased at the applied voltage of 24 kV (figures 2(i)–(l)). The rough fibers were formed due to buckling instability [56] and stretching by electrical force [42]. At the applied voltage higher than 18 kV, the jet will not be subjected to sufficient bending instability resulting in less roughness of fibers.
Table 1. Summarizing the polymer solution, processing parameters, and fibers diameter of samples electrospun at different levels of the applied voltage.

| Polymer solution | Applied voltage (kV) | DTC (cm) | Flow rate (ml h⁻¹) | Collector speed (rpm) | Needle diameter (mm) | Fibers diameter (nm) |
|------------------|----------------------|----------|--------------------|-----------------------|----------------------|---------------------|
| 14% (w/v)ACE     | 6                    | 18       | 1.5                | 2                     | 0.4                  | 969 ± 128           |
| 14% (w/v)ACE     | 12                   | 18       | 1.5                | 2                     | 0.4                  | 822 ± 105           |
| 14% (w/v)ACE     | 18                   | 18       | 1.5                | 2                     | 0.4                  | 761 ± 67            |
| 14% (w/v)ACE     | 24                   | 18       | 1.5                | 2                     | 0.4                  | 890 ± 78            |
| 27% (w/v)(ACE/DMF) | 6              | 18       | 1.5                | 2                     | 0.4                  | 1410 ± 198          |
| 27% (w/v)(ACE/DMF) | 12             | 18       | 1.5                | 2                     | 0.4                  | 1347 ± 176          |
| 27% (w/v)(ACE/DMF) | 18             | 18       | 1.5                | 2                     | 0.4                  | 1300 ± 155          |
| 27% (w/v)(ACE/DMF) | 24             | 18       | 1.5                | 2                     | 0.4                  | 1399 ± 188          |
| 22% (w/v)DMF     | 6                    | 18       | 1.5                | 2                     | 0.4                  | 743 ± 66            |
| 22% (w/v)DMF     | 12                   | 18       | 1.5                | 2                     | 0.4                  | 692 ± 57            |
| 22% (w/v)DMF     | 18                   | 18       | 1.5                | 2                     | 0.4                  | 650 ± 43            |
| 22% (w/v)DMF     | 24                   | 18       | 1.5                | 2                     | 0.4                  | 711 ± 60            |

It is worth mentioning that the diameter of fibers formed at different secondary surface morphologies alerted by maneuvering the applied voltage due to the changed in the electric field applied as shown in table 1 and figure S1 is available online at stacks.iop.org/MRX/7/015008/mmedia.

3.2. Effect of the flow rate on the secondary surface morphology of PVDF fibers

Flow rate, the amount of solution provided per unit time, is also a main parameter in the electrospinning process. Herein, to explore the relationship between the flow rate and the secondary surface morphology of PVDF fibers, PVDF fibers were electrospun at different flow rates while other processing parameters were fixed as shown in table 2.

For porous fibers, the fibres formed at a low flow rate has a bigger macroporous with elliptical shape than the fibres formed at a high flow rate (figures 3(a)–(c)). At a low flow rate, the water droplets on the jet’s surface have enough time to combine before reaching the collector. At the same time, these jets have enough time to evaporate their solvents before reaching the collector (high bending instability) resulting in forming elliptical pores [5].

For grooved fibers, it was found that there is a negative relationship between the flow rate and the number of grooves formed and that should be attributed to the decreased in the number of pores at the high flow rate (figures 3(d)–(i)) [55].

For rough fibers, the roughness degree of the fibers’ surface decreased by increasing the flow rate owing to decreasing the bending instability of the jet (figures 3(g)–(i)) [57].
Importantly, the diameter of fibers formed at different secondary surface morphologies increased by raising the flow rate as shown in table 2 and figure S3.

### 3.3. Effect of the distance between the tip of the needle and the collector (DTC) on the secondary surface morphology of PVDF fibers

Herein, PVDF solutions were electrospun at different DTC while other processing parameters were kept constant as shown in table 3.

For porous fibers, by increasing the DTC from 8 cm to 24 cm, the dimension of macroporous on the surface of fibers increased and their shape altered to elliptical (figures 4(a)–(c)). These results should be attributed to the increase in the jet flight time as well as decreased the electric field strength by increasing the DTC [6].

For grooved fibers, the number of grooves on the surface of fibers decreased by increasing the DTC from 6 cm to 24 cm and that should be ascribed to the decreased in the number of pores at long DTC (figures 4(d)–(f)).

### Table 2. Summarizing the polymer solution, processing parameters, and fibers diameter of samples electrospun at different levels of flow rate.

| Polymer solution          | Applied voltage (kV) | DTC (cm) | Flow rate (ml h⁻¹) | Collector speed (rpm) | Needle diameter (mm) | Fibers diameter (nm) |
|---------------------------|----------------------|----------|--------------------|-----------------------|----------------------|----------------------|
| 14% (w/v) ACE             | 18                   | 18       | 1                  | 2                     | 0.4                  | 649 ± 44             |
| 14% (w/v) ACE             | 18                   | 18       | 2                  | 2                     | 0.4                  | 823 ± 78             |
| 14% (w/v) ACE             | 18                   | 18       | 2.5                | 2                     | 0.4                  | 1014 ± 82            |
| 27% (w/v)(ACE/DMF)        | 18                   | 18       | 1                  | 2                     | 0.4                  | 1213 ± 144           |
| 27% (w/v)(ACE/DMF)        | 18                   | 18       | 2                  | 2                     | 0.4                  | 1399 ± 166           |
| 27% (w/v)(ACE/DMF)        | 18                   | 18       | 2.5                | 2                     | 0.4                  | 1523 ± 172           |
| 22% (w/v) DMF             | 18                   | 18       | 1                  | 2                     | 0.4                  | 598 ± 31             |
| 22% (w/v) DMF             | 18                   | 18       | 2                  | 2                     | 0.4                  | 725 ± 57             |
| 22% (w/v) DMF             | 18                   | 18       | 2.5                | 2                     | 0.4                  | 936 ± 69             |

![Images](image.png)

**Figure 3.** Representative SEM images of secondary surface morphology of PVDF fibers electrospun at different levels of flow rate. (a), (d), (g) 1 ml h⁻¹, (b), (e), (h) 2 ml h⁻¹, (c), (f), (i) 2.5 ml h⁻¹.
Regarding rough fibers, the roughness degree of the fibers’ surface increased by increasing the DTC from 6 cm to 24 cm and that should be ascribed to subjecting the flight jet to enough bending instability and electric force (figures 4 (g)–(i)) [6].

It was found that the diameters of porous, grooved, and rough fibers were decreased by increasing the DTC from 6 cm to 18 cm, however, the increase in the diameter of fibers at the DTC of 24 cm because the electric field force is not enough to provide sufficient stretch to flight jet.

### 3.4. Effect of the needle diameter on the secondary surface morphology of PVDF fibers

Different researches about the effect of the needle diameter on the surface morphology of electrospun fibers were done [58–61]. However, the effect of the needle diameter on the secondary surface morphology of electrospun fibers has not been studied.

Herein, PVDF solutions were electrospun at different needle diameters while other processing parameters were fixed as shown in table 4.

![Figure 4](image)

**Figure 4.** Representative SEM images of secondary surface morphology of PVDF fibers electrospun at different levels of DTC. (a), (d), (g) 8 cm. (b), (e), (h) 13 cm. (c), (f), (i) 24 cm.

| Polymer solution | Applied voltage (kV) | DTC (cm) | Flow rate (ml h⁻¹) | Collector speed (rpm) | Needle diameter (nm) | Fibers diameter (nm) |
|------------------|----------------------|----------|--------------------|-----------------------|----------------------|----------------------|
| 14% (w/v) ACE    | 18                   | 8        | 1.5                | 2                     | 0.4                  | 1031 ± 111           |
| 14% (w/v) ACE    | 18                   | 13       | 1.5                | 2                     | 0.4                  | 889 ± 97             |
| 14% (w/v) ACE    | 18                   | 24       | 1.5                | 2.0                   | 0.4                  | 693 ± 58             |
| 27% (w/v) ACE/DMF| 18                   | 8        | 1.5                | 2                     | 0.4                  | 1522 ± 181           |
| 27% (w/v) ACE/DMF| 18                   | 13       | 1.5                | 2                     | 0.4                  | 1411 ± 163           |
| 27% (w/v) ACE/DMF| 18                   | 24       | 1.5                | 2                     | 0.4                  | 1239 ± 119           |
| 22% (w/v) DMF    | 18                   | 8        | 1.5                | 2                     | 0.4                  | 930 ± 72             |
| 22% (w/v) DMF    | 18                   | 13       | 1.5                | 2                     | 0.4                  | 756 ± 56             |
| 22% (w/v) DMF    | 18                   | 24       | 1.5                | 2                     | 0.4                  | 595 ± 38             |

Regarding rough fibers, the roughness degree of the fibers’ surface increased by increasing the DTC from 6 cm to 24 cm and that should be ascribed to subjecting the flight jet to enough bending instability and electric force (figures 4 (g)–(i)) [6].

It was found that the diameters of porous, grooved, and rough fibers were decreased by increasing the DTC from 6 cm to 18 cm, however, the increase in the diameter of fibers at the DTC of 24 cm because the electric field force is not enough to provide sufficient stretch to flight jet.
secondary surface morphology of electrospun fibers. However, the diameter of fibers increased by increasing the needle diameter and that should be attributed to the kinetic energy of polymer chains. Increasing needle diameter leads to a decrease in the kinetic energy of the polymer solution. Reducing the kinetic energy of polymer chains leads to decreased whipping and bending during electrospinning [62]. Therefore, the decrease of polymer chains whipping and bending resulting in increasing the average diameter of nanofibers as shown in table 4 and figure S4.

3.5. Effect of the rotation speed of the drum collector on the secondary surface morphology of PVDF fibers

Herein, PVDF solutions were electrospun at different rotation speeds of the drum collector while other processing parameters were kept constant as shown in table 5.

Previous studies proved that the alignment and orientation degree of PVDF nanofibers increased by increasing the rotation speed of the drum collector owing to the mechanical and shear force exerted by the rotation speed of the drum collector rotation [1, 63, 64]. However, so far no studies have been reported the effect of the rotation speed of the drum collector on the secondary surface morphology of electrospun fibers.
The results showed that by increasing the rotation speed of the drum collector from 1000 rpm to 3000 rpm, the shape of macroporous on the surface of fibers altered to elliptical (figures 6(a)–(c)). Moreover, the deep of the grooves on the surface of fibers increased by raising the rotation speed of the drum collector (figures 6(d)–(f)). Furthermore, the roughness of fibers increased by raising the rotation speed of the drum collector (figures 6(g)–(i)). These results should be attributed to subjecting the fibers to high stretch at a high rotation speed of the drum collector and that also hence decreasing diameter of the fibers as shown in table 5 and figure S5 [1, 63].

### 4. Conclusion

In summary, we have studied the effect of processing parameters (applied voltage, flow rate, DTC, diameter of the needle, and rotation speed of the drum collector) on the secondary surface morphology of electrospun PVDF fibers. Porous fibers were obtained using 14% (w/v) ACE, while rough fibers were generated using 22% (w/v) DMF, whereas grooved fibers were formed using 27% (w/v) (ACE/DMF) (4:1) and all of them were electrospun.
at the relative humidity of 65%. The results showed that the secondary surface morphology of fibers was changed by raising the applied voltage from 6 kV to 24 kV. Furthermore, maneuvering the flow rate (1 ml h⁻¹, 1.5 ml h⁻¹, 2 ml h⁻¹, and 2.5 ml h⁻¹), DTC (6 cm, 12 cm, 18 cm, and 24 cm), rotation speed of the collector (2 rpm, 1000 rpm, 2000 rpm, and 3000 rpm) has the ability to alters the surface structures of fibers formed. Nevertheless, it was found that there is no relationship between the diameter of the needle (0.1 mm, 0.4 mm, and 0.83 mm) and the secondary surface morphology of electrospun fibers. We believe this work can be used as a useful guideline for researchers in both the academic and industrial fields owing to the importance of the secondary surface morphology of fibers.

**Disclosure statement**

No potential conflict of interest was reported by the authors.

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