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Fabrication and characterization of forcespun polycaprolactone microfiber scaffolds

Deepa Kodali, Farooq Syed, Shaik Jeelani and Vijaya K Rangari ©

Department of Materials Science and Engineering, Tuskegee University Tuskegee, AL 36088, United States of America

E-mail: vrangari@tuskegee.edu

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Abstract

Forcespinning technique was used to fabricate sub-micron size polycaprolactone (PCL) fibers. Forcespinning method uses centrifugal forces for the generation of fibers unlike the electrospinning method which uses electrostatic force. PCL has been extensively used as scaffolds for cell regeneration, substrates for tissue engineering and in drug delivery systems. The aim of this study is to qualitatively analyze the force spun fiber mats and investigate the effect of the spinneret rotational speed on the fiber morphology, thermal and mechanical properties. The extracted fibers were characterized by scanning electron microscopy differential scanning calorimetry, tensile testing and dynamic mechanical analysis. The results showed that higher rotational speeds produced uniform fibers with less number of beads. The crystallinity of the fibers decreased with increase in rotational speeds. The Young’s modulus of the forcespun fibers was found to be in the range of 3.5 to 6 MPa. Storage and loss moduli decreased with the increase in the fiber diameter. The fibers collected at farther distance from spinneret exhibited optimal mechanical properties compared to the fibers collected at shorter distances. This study will aid in extracting fibers with uniform geometries and lower beads to achieve the desired nanofiber drug release properties.

1. Introduction

The production of nonwoven fiber mats has recently gained considerable attention due to their potential applications in filtration, composite reinforcements, sensors, tissue scaffolding and many others [1–6]. The ideal scaffolds for bone regeneration and tissue engineering not only require optimal mechanical properties but also should have biocompatibility and biodegradability promoting the cell growth, adhesion and proliferation [7, 8]. Nano fibrous membranes with their unique physiochemical characteristics are highly capable of mimicking extracellular matrices and are ideal for bone regeneration and tissue engineering [9, 10]. Additionally, the polymer fiber mats exhibit versatile characteristics such as high surface area, high porosity and wide range of pore sizes [11]. Therefore it is of great importance to develop bioactive nanofibrous scaffolds with desirable mechanical properties.

Several methods such as self-assembly, phase separation, template synthesis, melt blowing and electrospinning was used to produce fibers. Among these various methods, electrospinning is the most commonly used method for the production of micro and nanofibers [4, 12–14]. This method uses high voltage electrostatic forces between the electrically charged polymer solution and the conductive collectors to generate fibers [15]. Substantial experimental work has been done on electrospinning with different types of polymers [16]. Despite being flexible and laboratory efficient for microfiber production, the electrospinning method is not suitable for industrial production due to its limitations such as usage of high-voltage electrical fields, low fiber yields, sensitivity to solution conductivity, lack of control over fiber orientation, difficulty in fabrication of three dimensional structures and environmental concerns [16, 17]. Although various strategies have been developed over the past two decades to advance the electrospinning methods, persistence of significant challenges due to dearth of high production demands reliable methods to generate well oriented and organized polymer microfibers. [4, 14]
However, recently introduced forcespinning technique was able to overcome the disadvantages that were encountered by electrospinning method. Forcespinning uses centrifugal force to generate the fibers and hence, the restrictions imposed on the dielectric materials through electrospinning have been eliminated [4, 18]. As a result, the forcespinning method can be used for a wide variety of conductive and nonconductive polymers, solutions and melts without using electric fields [19]. Furthermore, the yield obtained from forcespinning (50–100 g h⁻¹) at lab scales is reported to be higher than electrospinning (0.5 g h⁻¹) [4, 18, 20]. The morphology of the fibers produced from the forcespinning depends on the controllable factors such as angular velocity of the spinneret, orifice radius, viscosity of the solution, surface tension, evaporation rate of the solvent and the distance between the collector and the spinneret [21, 22]. Forcespun fibers were successfully produced using polymers such as polyactic acid, polyvinyl alcohol and polycaprolactone [4, 18, 23].

Specifically, PCL is a biodegradable polymer with good biocompatibility and thermal stability that has been widely used in both industrial and biomedical applications [24]. The exceptional mechanical properties combined with the biodegradability made PCL an undeniable source of application in tissue engineering, dental splints, drug delivery systems and constituent materials in nanocomposites [10, 25]. In view of the extensive applications of the PCL, it is highly important to understand and contribute to the methods that will generate higher production of PCL fibers. Also, it is important to understand the micro mechanical properties of the fibers to ensure the performance of these fibrous scaffolds for biomedical applications.

The objective of this present study was to investigate the effects of rotational speed on the morphology, crystallinity, thermal and mechanical properties of the PCL fibers. The tests were carried out by varying rotational speeds for the PCL solution. Based on the studies by Van der Schueren and her co-workers [26], chloroform resulted in uniform and thick fibers compared to the other solvents like acetic acid and formic acid. Hence, in the present study chloroform was used as a solvent for preparing PCL solution. The produced fibers were analyzed using scanning electron microscopy (SEM) and differential scanning calorimetry (DSC). The mechanical and dynamic mechanical properties of the PCL fibers were analyzed using tensile tests and dynamic mechanical analysis (DMA). Furthermore, the effect of the distance between the spinneret and the collector plate was also investigated.

2. Materials and methods

2.1. Materials

The powdered PCL with a molecular weight of 50000 was purchased from Polysciences, Inc. (Warrington, PA, USA). The solvent chloroform with ACS reagent ≥ 99.8% was purchased from Sigma-Aldrich (St. Louis, MO, USA).

The PCL solution was prepared by dissolving the powdered PCL in chloroform in concentration of 16 wt%. The PCL solution was then mixed using a magnetic stirrer for three hours at 170 rpm. The vials were sealed during the mixing process to prevent the evaporation of solvent.

2.2. Forcespinning of fibers

The nonwoven fibrous mats were obtained from the polymer solution mixture using forcespinning Cyclone L-1000M apparatus from Fiberio which was equipped with solution spinneret with 27ga × ½’’ stainless steel regular bevel needle. Using a syringe, a 2 ml of the precursor solution was injected into the spinneret. Fibers were collected at rotational speeds of 5000, 7000 and 9000 rpm with a spin time of 10 min. A fiber collector with equally spaced vertical plates placed at a distance of 115 mm from the needle (190 mm from the center of the spinneret) was used to collect the fibers as shown in figure 1(a). Finally, the collected fibers are shown in figure 1(b) were stored under desiccation prior to the characterization.

2.3. Fiber characterization

2.3.1. Scanning electron microscopy

The morphology of the fibers obtained for three different rotational speeds was analyzed using JEOL JSM-7200F field emission scanning electron microscope (FESEM, JEOL USA, Peabody, MA) at 2 kV. The samples were sputter coated with gold/palladium (Au/Pd) for 3 min at 10 mA using Hummer sputter coater.

2.3.2. Differential Scanning Calorimetry

The thermal properties of the fibers were analyzed using DSC TA-Q series 2000. The samples weighing 10 to 12 mg approximately were sealed using hermetic pans. The DSC thermograms were obtained at a rate of 5 °C min⁻¹ from −80 °C to 80 °C followed by cooling to −80 °C and then heating up to 80 °C.
2.3.3. Tensile testing
The uniaxial tensile tests were performed to analyze the mechanical properties of the nonwoven fibrous mats with 5 mm width and 20 mm length following ASTM D882-10 standard [27]. The test window frame with fibrous mat is shown in figure 2(a). The thickness was measured with micrometer (Mitutoyo 293-340-30 digital micrometer) with 0.001 mm resolution. To obtain statistically reliable results, the average thickness of the fibrous mats was obtained by taking measurements at 10 different places.

Finally, the test window frame was placed between the grips of Zwick/Roell Z2.5 universal mechanical testing machine and the frame portion was cut as shown in figure 2(b). Displacement control mode with constant crosshead speed of 5 mm min\(^{-1}\), preload of 0.01 N, and a 20 N load cell was used to perform the tensile tests (figure 2(c)). The Zwick/Roell software associated with the machine calculates the slope of each stress-strain curve in its elastic deformation region to obtain the Young’s modulus. Additionally, the software also gives other desired test results like elongation at break, tensile strength, elongation at maximum stress.

2.3.4. Dynamic mechanical analysis
The samples for DMA were cut from the fibrous mats with size of 40 mm × 5 mm. The thickness was measured with Mitutoyo digital micrometer with 0.001 mm resolution at ten different places and was then averaged to obtain the mean thickness of the mat. The dynamic mechanical behavior of the fibrous mats was characterized.
using dynamic mechanical analyzer TA-Q series 800 in tensile mode under multifrequency strain mode (frequency sweep method) with a static preload of 0.01 N. The specimen was then held isothermally at 30 °C oscillating at a constant strain of 1% over a frequency range of 1 Hz to 10 Hz.

3. Results

3.1. Characterization of PCL fibers
The SEM micrographs of the forcespun PCL fibers with 16 wt% concentration is shown in the figure 3. The fibers obtained were homogenous forming three-dimensional mesh with random orientation. There were no fibers extracted at less than 3000 rpm and fibers with large number of beads at 3000 and 4000 rpm respectively. Hence, in this study fibers obtained at 5000, 7000 and 9000 rpm were considered. As the rotational speed increased the fibers became more homogenous and the beading of the fibers decreased significantly. However, at high rotational speeds of 9000 rpm the diameter of the fibers decreased significantly, and very thin microfiber webs which resulted in the formation of intermittent fibers. The beading of the fibers was influenced by the solidification time between the ejection of polymer solution from the spinneret and the deposition of the solution on to the collector.

The relationship between the rotational speed of the spinneret and the fiber diameters is shown in table 1. The fiber diameter is obtained by following the Gaussian distribution. For the rotational speeds of 5000 rpm the fiber diameter is 1.98 μm. As the rotational speed increases to 7000 rpm the fiber diameter decreases. For 9000 rpm the diameter of the fibers was reduced significantly. The results in table 1 suggest that with the increase in rotational speed the fiber diameter decreases. As the rotational speed of the spinneret increases, the orbital trajectory that the fibers travel along expands. During this process, the fibers become more stable and elongate resulting in the reduction of diameter. In addition, the polymer jet experiences Coriolis force, drag force, centrifugal force and viscous forces. As the speed of the spinneret increases, the aerodynamic forces caused by the spinneret increases due to the vibrations produced by the fibers which results in reduction of the diameter of the fibers [22]. However, the entirety of the diameter does not depend solely on the rotational speed but also

| Rotational speed (rpm) | Mean fiber diameter (μm) |
|------------------------|--------------------------|
| 5000                   | 1.98±0.709               |
| 7000                   | 1.65±0.173               |
| 9000                   | 1.4±1.393                |

Figure 3. SEM micrographs of PCL fibers with corresponding histograms showing fiber diameter distribution for concentration of 16 wt% at various rotational speeds of the spinneret (a) 5000 rpm (b) 7000 rpm and (c) 9000 rpm.
other important controlling factors such as solidification time and collection time of fibers after deposition. Moreover, orifice radius and orientation, fluid viscoelasticity, surface tension, solvent evaporation rate, fluid fill level, and distance of the collector plates to the spinneret also play an important role in influencing the diameter of the generated fibers [21, 22].

3.2. DSC analysis

The thermal properties of the PCL fibers were analyzed from the DSC thermographs as shown in figure 4. The melting ($T_m$), crystallization ($T_c$) temperature, enthalpy ($\Delta H_m$) and enthalpy of crystallization ($\Delta H_c$) were evaluated from the second heating cycle of the DSC endotherms and were tabulated in table 2. The glass transition temperature slightly decreased with the increase in the rotational speed (figure 4(a)). The fibers at 5000 rpm have higher glass transition temperature of $−57.57^\circ$. The peak melting points of the fibers do not show any pivotal shift with the increase in the rotational speed as shown in figure 4(b). The enthalpy (melting and crystallization) vary slightly with the increase in rotational speed (figures 4(b) and (c)).

The crystallinity was calculated from the following equation:

$$\chi_c = \frac{\Delta H_m - \Delta H_f}{\Delta H_f},$$

where $\chi_c$ is crystallinity and $\Delta H_f$ is the enthalpy of fusion of 100% crystalline sample (139.5 J g$^{-1}$ for PCL). The crystallinity of the fibers is shown in table 2. The morphology of the fibers is strongly influenced by the solvent which allows for chain relaxation. The crystallinity decreased with the increase in the rotational speeds similar to the results reported by McEachin et al [4] which might be due to incomplete crystallization [28]. Owing to the short crystallization time, the crystals formed are small in size with defects which resulted in the low melting point of fibers and decrease in crystallinity [29]. The second peaks for melting endotherm were present similar to the results presented by McEachin et al [4].

3.3. Tensile test analysis

Typical stress–strain curves for the forcespun microfiber mats obtained at 5000, 7000, and 9000 rpm were shown in figure 5. Average of the four samples was considered for each rotational speed. The results obtained from the tensile test of the forcespun PCL fiber mats were tabulated in table 3. The fibers at 5000 rpm have high Young’s modulus of 5.08 MPa and 9000 rpm have least Young’s modulus of 3.89 MPa. The Young’s modulus decreased with the decrease in crystallinity. The tensile strength of the fibers decreased with increase in rotational speeds which can be attributed to the decrease in glass transition temperature and crystallinity. Furthermore, with the decrease in the diameter of the fibers there might be high porosity within the fibrous membranes which resulted in the decrement of the Young’s modulus and tensile strength. The overall Young’s Modulus of the fiber

![Figure 4](image_url) (a) DSC thermographs showing glass transition temperature, (b) melting endotherms and (c) crystallization temperatures for PCL fibers for various rotational speeds.

Table 2. DSC analysis for the PCL fibers obtained at various rotational speeds.

| Rotational speed (rpm) | Glass transition temperature | Heating | Cooling |
|------------------------|------------------------------|---------|---------|
|                        | $T_g$($^\circ$C) | $T_m$($^\circ$C) | $\Delta H_m$ (J g$^{-1}$) | $T_c$($^\circ$C) | $\Delta H_c$ (J g$^{-1}$) | % Crystallinity ($\chi_c$) |
| 5000                   | $−57.57$            | 60.01   | 83.06   | 32.25   | 81.54       | 1.09               |
| 7000                   | $−59.71$            | 59.81   | 83.66   | 30.02   | 82.41       | 0.9                |
| 9000                   | $−61.72$            | 59.74   | 83.27   | 31.29   | 82.46       | 0.58               |
mats was observed to be in the range of 3.5 to 6 MPa which conforms to the results that were reported for the electrospun PCL fibers (3.5–6 MPa) [30, 31]. The ductility of the stress strain curves can be attributed to the low glass transition temperatures of around $-60^\circ$C [32]. The tensile tests were conducted at room temperature which is far higher than that of the glass transition temperature of PCL fibers. As a result, mobility in molecular chains might have increased causing the disentanglement in the chains thus allowing them to mobilize at low loads. The mean value of strain at break was observed as 150%–190% [30, 33]. In the present study, the strain at break was observed to be ranging from 215%–375% which was very high compared to the reported results [30, 33]. This suggests that the stretchability of the fibers had increased from force spinning technique.

### 3.4. DMA test analysis

The viscoelastic properties of the fibrous membranes were analyzed using dynamic mechanical analyzer which correlates the strain induced by the application of constant dynamic stress [34]. Figure 6 shows the viscoelastic behavior of the specimens over a specified frequency range of 1 Hz to 10 Hz. The storage modulus of the fiber mats increases with the increase in frequency and decreases with the rotational speed as shown in figure 6(a). Although there is no significant change in the storage modulus, PCL fiber with 9000 rpm showed the least modulus compared to others. The decrease in the storage modulus suggests that the elastic response varies with the networking structure of the fibers. This also can be attributed to the crystallinity of the fibers which suggests that the fibers with misaligned molecular structures due to short crystallization times. The loss modulus decreases with the increase in frequency as shown in figure 6(b).

### 3.5. Fiber characteristics at various collector plate distances

To further analyze the fiber characteristics, a study was conducted based on the collector plate distance from the spinneret. From the previous results, it was observed that PCL with 5000 rpm (collected at 190 mm from the center of the spinneret) exhibits better mechanical properties compared to fibers at 7000 and 9000 rpms. Hence, PCL fibers were collected at 5000 rpm for two collector plate distances i.e., 165 mm and 270 mm. The results for the fiber analysis were tabulated in table 4. The mean diameter of the fibers collected at 165 mm from the center of the spinneret was 2.03 μm which is slightly higher than the diameter of the fibers collected at 190 mm. As the

![Figure 5. Averaged stress-strain curves of PCL microfiber mats obtained at 5000, 7000 and 9000 rpm.](image)

Table 3. Data summary of the tensile test analysis for force spun PCL fibers at various rotational speeds.

| Rotational speed (rpm) | Young’s Modulus (MPa) | Tensile strength (MPa) | Strain(%) at break |
|------------------------|-----------------------|-----------------------|-------------------|
| 5000                   | 5.08 ± 1.48           | 1.34 ± 0.42           | 375               |
| 7000                   | 4.7 ± 0.81            | 1.07 ± 0.19           | 214               |
| 9000                   | 3.89 ± 0.61           | 1.02 ± 0.41           | 363               |
The distance between the spinneret and the collector plate increases the diameter of the fiber decreases. The fibers collected at 270 mm from the spinneret have a fine diameter of 0.91 μm as shown in figure 7(a).

The crystallinity of the fibers increased with the increase in the collector plate distance. The fibers collected at 270 mm show high crystallinity of 1.33% compared to the other fibers as shown in figure 7(b). This can be attributed to the fine diameter of the fibers which allowed for crystallization before deposition of the jet on the collector plates following the higher degree of molecular orientation.

The tensile properties of the fibers were shown in figure 7(c). Four samples were tested for tensile properties and the average of the results was considered. As the crystallinity increases, the Young’s modulus and the tensile strength increases. The fibers collected at 270 mm distance have high tensile strength of 1.8 MPa as shown in table 4. However, the elongation decreases for the fibers with fine diameter. As the fibers collected at 270 mm have higher degree of molecular orientation compared to the fibers collected at 190 mm, the ductility of the fiber’s decreases. Although the fibers collected at 165 mm were thick and have lower crystallinity the elongation is less compared to the fibers collected at 190 mm. This might be due to the imperfections in the molecular orientation which tends to break the fibers soon. The viscoelastic properties of the fibers collected were shown in figure 7(d). The storage modulus of the fine fibers collected at 270 mm is higher than that of the other fibers and increases with increase in the frequency. This suggests that the stiffness of the fibers increases with the higher degree of molecular orientation.

| Collector plate distance (mm) | Mean fiber diameter (μm) | Crystallinity % | Young’s modulus (MPa) | Tensile strength (MPa) | Strain at break (%) |
|-------------------------------|-------------------------|----------------|-----------------------|-----------------------|--------------------|
| 165                           | 2.03                    | 1.03           | 4.4 ± 2.36            | 1.12 ± 0.44           | 297                |
| 190                           | 1.98                    | 1.09           | 5.08 ± 1.48           | 1.34 ± 0.42           | 375                |
| 270                           | 0.91                    | 1.33           | 5.89 ± 1.54           | 1.8 ± 0.31            | 360                |

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4. Conclusions

Forcespinning technique was successfully used to produce the PCL fibers and the influence of the rotational speeds on the diameter of the fibers was also analyzed. The fibers at high rotational speeds have less beads suggesting that the degree of beading can be controlled by the rotational speed. The crystallinity and glass transition temperature decreased with higher rotational speeds. Also, at higher rotational speeds, the reduced fiber diameter might have resulted in the induced mesophase with incomplete crystallization. The tensile strength of the fibrous mats decreased with increased rotational speeds due to the reduced crystallinity. The fibers collected have random orientation although the fibrous mats were prepared in the stretching direction. However, the mechanical properties observed from the tensile tests of the fibrous mats were in good agreement with the results reported in literature. The study conducted based on the collector plate distance suggests that increasing the distance between the spinneret and the collector plate also allows the jet to be crystallized before

Figure 6. (a) Storage modulus and (b) loss modulus of PCL microfiber mats for various rotational speeds and frequencies.

Table 4. Data summary of PCL fibers collected at various distances from the spinneret for 5000 rpm.
deposition which is followed by a higher degree of molecular orientation of the fibers. The mechanical properties of the fibers addressed in this study helps to understand the factors that highly influence the morphology of the forcespun fibers.

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ORCID iDs

Vijaya K Rangari @ https://orcid.org/0000-0002-3962-1686

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Figure 7. PCL fiber characteristics at various collector plate distances: (a) mean fiber diameter, (b) crystallinity, (c) tensile properties and (d) storage modulus.
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