Materials Letters 140 (2015) 99–102

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

# Identifying key processing parameters for the electrospinning of aligned polymer nanofibers

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ARTICLE INFO

*Article history:*
Received 25 June 2014
Accepted 29 October 2014
Available online 6 November 2014

*Keywords:*
Electrospinning
Fiber alignment
Polymers
Fiber technology
Porous materials

ABSTRACT

Aligned polycaprolactone (PCL) fibers of interest for numerous applications requiring contact guidance and textured topography were fabricated by electrospinning onto a rotating collector using tangential velocities ranging from 0 m/s to 11 m/s. The deposition characteristics of the fibers were investigated by microscopic methods to gather information about fiber diameter, collection angle θ, and number of fibers per reference length. It was shown that the deposition angle ranged from −2.0° to 1.6° and the diameter of the developed fibers averaged 587 nm to 878 nm. Furthermore the deposition angle was found to be independent of the rotation velocity. Homogeneity of the fiber mats was improved with increasing rotation velocity. The number of fibers per reference length, a measure for collection efficiency, proved to be dependent on the rotation velocity, following a modified Weibull function. Hence the collection efficiency of the process can be improved by increasing the rotation velocity above a critical value.



## 1. Introduction

Electrospinning is a fiber technology being increasingly used to produce porous, fibrous structures for a wide range of applications involving filtration devices, biomedical scaffolds, composites and applications in the energy and environmental sectors [1,2]. Aligned fibers are particularly interesting for biomedical applications as they can mimic the aligned structure of some tissues, thus providing contact guidance and orientation to cells [3,4]. Aligned fibers are also of relevance for several technological applications such as water filtration, functional nanocomposites and energy conversion devices [5,6].

The creation of aligned fibers is a more recent development in electrospinning which started at the beginning of the 21st century. Aligned fibers can be obtained by different means, for example, using a rotating mandrel [7,8], a rotating disk [9], or employing non-solid rotating devices such as a rotating circular drum with copper wires [10], or metal frame collectors [11] among other approaches. To date, a large number of reports concerning different techniques to align fibers and additional alterations of the process, such as auxiliary electrodes to create a focusing force [12], have been published. However, the specifics of the alignment process have received only limited systematic research efforts as yet.

In this study a rotating collector was used to produce aligned polymer fibers. Polycaprolactone (PCL) was chosen due to its significance in biomedical applications [13] but the results can be extended for other polymers. Relevant information about the collection angle θ (angle between fiber and machine direction), the number of fibers collected per reference length (collection efficiency) and the fiber diameter was gathered. As stated above no systematic study varying the processing parameters for obtaining aligned fibers by electrospinning has been undertaken so far, hence the analysis of the results obtained in this study is crucial to gain in depth knowledge about the process.

## 2. Experimental work

### 2.1. Materials

Polycaprolactone (PCL) with an average molar mass of 48–90 kDa was purchased from Aldrich, Germany. Dichloromethane (DCM) and methanol were used as solvents, which both had a density of 1.33 g/cm³ and were analytical grade reagents (Sigma, Germany).

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http://dx.doi.org/10.1016/j.matlet.2014.10.150
0167-577X/

### 2.2. Electrospinning setup

The setup consists of three main parts: a syringe delivering the polymer solution, a HV power supply (Linari SRL, Pisa Italy) and a rotating collector for the fibers, which was purpose-built at the University of Erlangen-Nuremberg. A 3D model of the rotating collector is shown in Fig. 1a. As shown in Fig. 1b the rotating collector consists of two circular terminal plates equipped with circular notches. The notches enable mounting of equally spaced horizontal bars. The bars are held in place by screws. In this study eight bars were used. The spacing between two adjacent bars is 70 mm. The rotating collector has a diameter of 21.2 cm. It can be used up to a speed of 1000 rpm, which corresponds to a tangential velocity of ≈ 11 m/s.

### 2.3. Fiber production and sample collection

A 10 wt % solution of PCL was obtained by completely dissolving the polymer in a mixture of DCM and methanol in a ratio of 7:3 by stirring at room temperature for 24 h prior to electrospinning to ensure a homogeneous solution. No solution older than 72 h was used. The solution was kept in an airtight container at ~ 7°C. A syringe mounted on a syringe pump dispenses the polymer solution. Its metallic tip has an inner diameter of 0.514 mm. The pump was operated at a flow rate of 4 ml/h. A voltage of 18 kV was used for the electrospinning process in this study and the distance between needle and collector was 15 cm.

The fibers were produced at velocities ranging from 0 to 1000 rpm. Fig. 1 summarizes experimental details used in the study. The PCL fibers were collected directly from the rotating collector. This was done using SEM mounts equipped with carbon tape, as shown in Fig. 1c, so that the fibers themselves and the orientation of the fibers were disturbed as little as possible.

### 2.4. Analysis of fiber alignment, diameter and collection efficiency

Samples were sputter coated for 1 min (35 V, 1.25 mA) at $3 \times 10^{-1}$ mbar with gold-palladium alloy to be viewed with the scanning electron microscope. The SEM used was a 435 V JEOL (Zeiss, Germany) and all images were taken using an accelerating voltage of 5 kV. SEM images were taken at magnifications of 500 × and 5000 ×. High magnification images were used to determine the fiber diameters. The images were evaluated using ImageJ (public domain, National Institutes of Health, USA, version 1.46r). The orientation angles of the fibers were measured against the vertical edge of the image because the vertical axis of the image had been positioned to coincide with the machine direction (=MD, see Fig. 1b). To obtain the alignment along the machine direction 90° were subtracted. To quantify the collection efficiency the number of fibers over a given distance was counted and given in count/μm. Table. 1 summarizes the parameters. The statistical analysis was carried out using Origin (OriginLab Corporation, USA, version 9.0.0 G). For constructing the histograms of deposition angles a bin size of ± 2.5° was chosen as suitable compromise to illustrate possible misaligned fibers.

## 3. Results and discussion

Fig. 2 shows the alignment and deposition angles for three different rotating speeds. They were chosen to reflect the range of speeds investigated: a) 0 rpm no rotation, b) 50 rpm slow rotation speed and c) 900 rpm high rotation speed. As can be seen in the photographs, in the left column, the amount of fibers collected on the rotating collector increased proportionally to the velocity of the collector. Moreover, the fibers became gradually more homogenous for the higher collection speeds since less debris was observed on the fibers, which might be due to a change in the electrostatic field between the needle tip and the collector due to the increased rotation speed.

The SEM images (Fig. 2, middle column) reveal that in the case of a stationary collector the majority of fibers were reasonably aligned and straight, but some fibers were neither aligned nor straight, which made it difficult to determine the deposition angle (a). For the fibers spun at 50 and 1000 rpm all fibers were straight and the majority were aligned in the machine direction (b, c). The histograms (Fig. 2, right column) show that the main deviation of deposition angle took place between −45° and +45°. The highest

**Table 1**
Summary of all test parameters and results including rotation speed, time, deposition angle, fiber diameter, number of fibers per reference length, and number of replications.

| speed | tangential velocity | time | deposition angle | | diameter | | fibers/ref. length | |
|---|---|---|---|---|---|---|---|---|
| rpm | m/s | min | ° | n | nm | n | 1/mm | n |
| 0 | 0 | 15 | −0.5 ± 20.8 | 102 | 878 ± 300 | 21 | 65.7 ± 10.3 | 7 |
| 10 | 0.1 | 120 | −2.0 ± 14.1 | 118 | 776 ± 251 | 29 | 122.9 ± 14.3 | 3 |
| 50 | 0.6 | 120 | −0.8 ± 13.5 | 162 | 664 ± 162 | 37 | 187.6 ± 29.3 | 3 |
| 100 | 1.1 | 120 | 1.6 ± 14.4 | 115 | 752 ± 119 | 13 | 207.6 ± 19.4 | 3 |
| 300 | 3.3 | 120 | 0.1 ± 12.7 | 149 | 665 ± 220 | 28 | 164.8 ± 17.2 | 3 |
| 900 | 10 | 60 | −1.4 ± 12.7 | 285 | 587 ± 133 | 45 | 674.3 ± 54.5 | 3 |
| 1000 | 11.1 | 60 | −1.7 ± 13.6 | 223 | 742 ± 230 | 26 | 558.1 ± 53.1 | 3 |

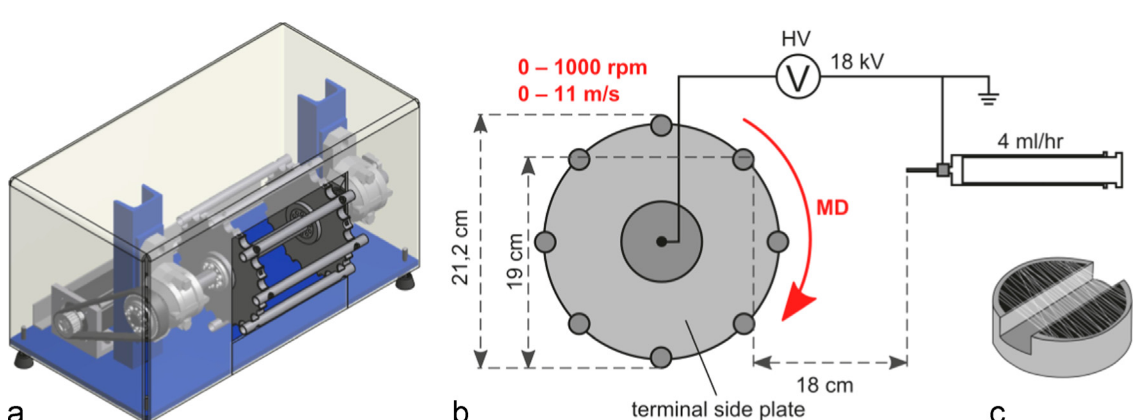


**Fig. 1.** Custom-made rotating collector. (a) 3D-model: Rotating collector which is driven by a v-belt (courtesy of Mr. Ossege). (b) Schematic of experimental setup with rotating collector and syringe pump, including settings and spatial arrangement. MD=machine direction. HV=high voltage. (c) SEM mount with carbon tape used to collect fiber samples.