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Electrospinning with dual collection rings

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Abstract

The formation of electrospun poly(ε-caprolactone) (PCL) fibres between two collection rings is described, and the conversion of these fibres into a multi-filament yarn of is demonstrated. During electrospinning, when two grounded rings are placed equidistantly from the spinneret, an array of fibres is formed between the collection rings. These rapidly produced fibres are three-dimensionally suspended in air, and demonstrate low incidence of fibre splitting when lower voltages are applied. Such electrospun fibres, with a diameter of 1.26 ± 0.19 μm and a length between 40 and 100 mm, were oriented and continuous. Furthermore, manufacture of the fibre array was easily achievable, reproducible and not subject to relatively small changes in spinneret-ground distance or applied voltage. Rotation of one of the collection rings results in a wound multi-filament yarn with a diameter below 5 μm, and a length of 50 mm.

Keywords: Electrospun scaffold; Tissue engineering; Suture

1. Introduction

Electrospinning has attracted recent interest as a technique for manufacturing sub-micron fibres for a variety of applications [1], including filtration, drug delivery systems and as substrates for cellular support [2–4]. Electrospun fibres, which are continuous in length, have diameters ranging from under 3 nm to over 2 μm in diameter, depending on the electrospinning conditions. For biomedical applications where cell-material interactions are of significance, electrospun fibres have a similar diameter range to that of fibrils in certain extracellular matrix [5], and have attracted interest as a cell-supporting substrate for tissue engineering.

For certain applications, it is of interest to electrospin alternative structures instead of the non-woven mats that are commonly formed from this process [6]. However, the configuration of the grounded collection plate for electrospinning has been limited, with most collections systems resulting in a thin sheet of fibres being formed. The vast majority of electrospinning systems use a single collection plate to collect the fibres (Fig. 1(a)) [2,4,7,8], while earthing a single rotating drum has commonly been employed to collect mats of this 'fabric' and to orient the fibres in this flat nanofibrous thin sheet (Fig. 1(b)) [9–11]. Oriented fibres can be obtained using dual, grounded collection plates, either next to a flat plate (Fig. 1(c)) or by itself [12], or with the fibres collected in a space between two rectangular collection plates (Fig. 1(d)) [13,14]. This collection process has been termed as the 'gap method of alignment', and it results in single electrospun fibres, oriented and suspended between two collection plates [15].

A progression with the gap method of alignment, described in this article and shown in Fig. 1(e), involves grounding two collection rings or circular disks equidistant from the spinneret. This permits the manufacture of fibres in a configuration different from nanofibrous sheets collected on a single plate, and leads to the development of suspended electrospun fibres with considerable volume. A simple, rapid and inexpensive technique to electrospin fibres arranged in an oriented, three-dimensional, configuration is herein described. Furthermore, by rotating one of the collection rings a multi-filament yarn consisting of

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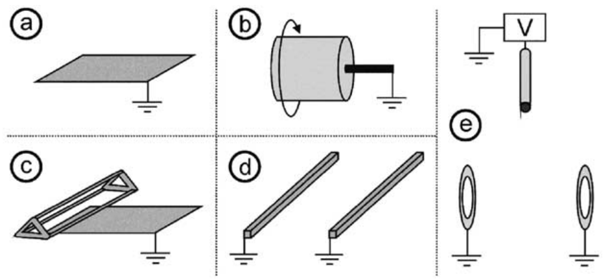

Fig. 1. Scheme of various electrospinning collection systems showing (a) single plate configuration, (b) rotating drum, (c) triangular frame placed near single plate, (d) parallel dual plate and (e) the dual-grounded ring configuration used in this article. The polymer solution is pumped to a spinneret (not shown in (a) to (d)).

electrospun fibres can be produced, demonstrating the ability to form more complex structures with electrospun fibres which were previously unattainable using current collection systems.

2. Materials and methods

2.1. Materials

All materials, unless otherwise stated, were purchased from Aldrich Chemicals (Milwaukee, WI, USA). Poly(ε-caprolactone) (PCL) was dissolved in a mixture of chloroform and methanol (75/25 v/v) to make a 9 wt% solution. The polymer solution was pumped to the 16-gauge, flat-tipped, stainless steel spinneret at a rate of 0.1 mL/h, through Teflon® tubing. Two stainless steel spacers (35 mm OD, 25 mm ID, 2 mm thick) were used as the collection rings and were positioned using shielded, grounded, alligator clips with the top of the rings being 150 mm from the spinneret. Unless specifically mentioned, the two rings were 80 mm horizontally apart. A voltage of 15 kV, unless otherwise specified, was applied to the polymer solution with a Series 205B high voltage power supply (Bertan, NY). All other surrounding metallic sources were shielded from the high voltage component while voltage was applied for 60 s.

2.2. Scanning electron microscopy (SEM)

The electrospun fibres were collected on an aluminium stub with parallel strips of carbon tape applied to the surface to promote fibre adhesion to the stub. After manufacture of the fibres, the stubs were passed through the suspended fibres between the collection rings. For SEM of the yarn, the formed fibre was collected and placed upon the specimen stub with tweezers. The samples were gold-coated (S150B Sputter Coater, Edwards), and imaged with SEM (Cambridge S360, Leica) using an accelerating voltage of 15 kV. Fibre and yarn diameters were calculated using SEM with $n=20$ measurements and are presented as average ± standard deviation. Representative images are presented.

2.3. Yarn manufacture

To manufacture a yarn, two grounded aluminium SEM stubs (12.5 mm diameter) covered with carbon tape were used as a collection system, with one of them attached to a Teflon® coated stainless steel tube, both grounded with copper wire. The outer part of the stubs was shielded with rubber septa, while the distance between collectors for this configuration was 60 mm. After collection of the electrospun fibres between the grounded disks using an applied voltage of 15 kV for 20 s, the stainless steel tube was inserted into a horizontally-mounted stirrer, and the copper wire removed from the ground assembly. The stirrer, and one collection disk, was rotated at 2500 rpm, until a single yarn was produced (Fig. 2). This typically took 60 s for the yarn to fully form between the collection rings.

3. Results and discussion

A series of fibres resulted between the two collection rings (Fig. 3), with the entire three-dimensional array of electrospun fibres being approximately 300 mL in volume. Formation of these fibres was easily achievable, reproducible and was not subject to relatively small changes in spinneret-ground distance or applied voltage.

From visual observations of the electrospun fibre formation, the first suspended fibres appear at the lower part of the collection rings (approximately 20 mm), between the shielded alligator clips. The fibre collection then proceeded upwards to the top of the ring, while the highest point of the collection rings was the last place were fibres were formed. Further electrospinning resulted in a dense distribution of fibres between the top points of the collection rings. When the collection rings were separated by a distance greater than 100 mm, the array of electrospun fibres was poor, and was heavily formed at the bottom of the collection rings between the shielded alligator clips. Reduction of the collection gap to below 40 mm also resulted in a poorly distributed collection of electrospun fibres; however, in this instance the fibres formed at the top of the collection rings.

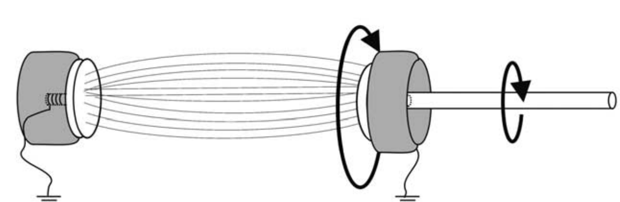

Fig. 2. Schematic of electrospinning collection system for fabricating a yarn of electrospun fibres. After formation of the fibres, the ground on the right is rotated, while the left ground remains stationary.

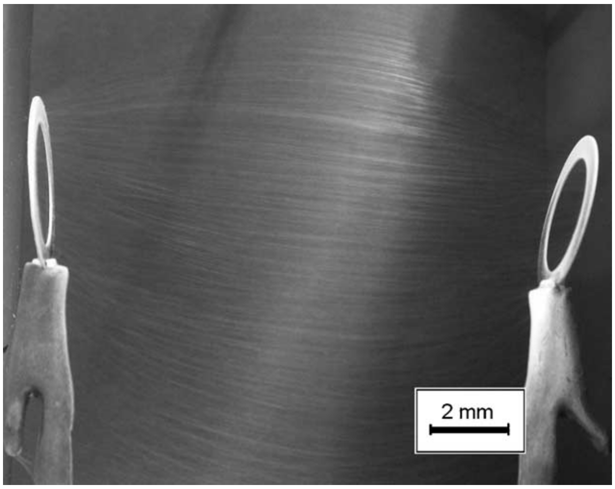

Fig. 3. Photograph of collection rings with an array of electrospun fibres, three-dimensionally suspended between them after 60 s of applied voltage.

Electrospun fibres formed from the application of 15 kV demonstrated a very low rate of fibre splitting (Fig. 4(a)), and the average diameter of the electrospun fibres was 1.26 ± 0.19 μm. The fibre diameter did not significantly alter when the distance between collection rings was varied between 40 and 100 mm. The fibres were generally oriented between the two clips; however, there was some cross-over of the fibres observed. Visual inspection of the fibres with a commercial laser pointer and with SEM determined them to bridge completely across the dual-collection system. The rings were coated with typical random electrospun fibres, as seen with single ground collection systems [2,4,7,8]. The collection of fibres has an outwards curvature immediately after formation, most likely due to an excess charge within the fibres (Fig. 3).

For electrospun fibres collected with the application of 25 kV, there was extensive splitting of the fibres, and the average fibre diameter was 0.85 ± 0.40 μm. The distribution of fibre sizes increased with higher voltages and the interconnectivity between the fibres was extensive (Fig. 4(b)). When a voltage of 30 kV was applied, there was poor formation of the electrospun fibre array, and the fibres were predominantly situated at the lower part of the collection system, between the shielded alligator clips.

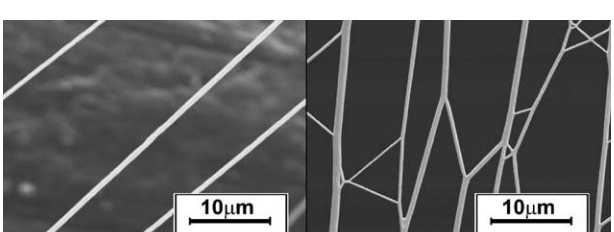

Fig. 4. SEM micrograph of electrospun polymers produced with (a) 15 kV and (b) 25 kV.

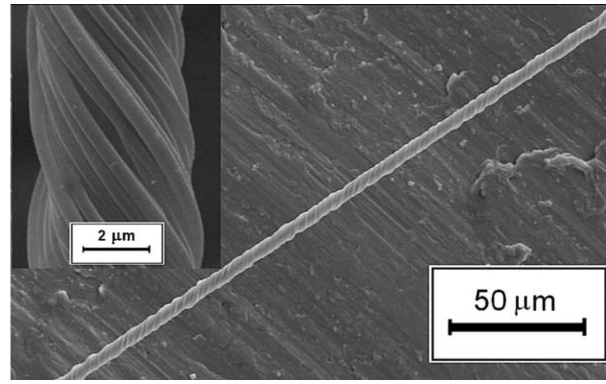

Fig. 5. SEM micrograph of wound PCL fibres, forming a multi-filament yarn. The insert shows a larger magnification of the yarn.

A yarn of PCL, 4.7 ± 0.4 μm in diameter ($n = 10$ measurements along the yarn length), is shown in Fig. 5 with the thickness influenced by the collection period of the fibres. Collection amounts above and below a certain range resulted in poor quality multi-filament yarns, indicating there is a maximum diameter that multi-filament yarns can be formed using this technique. The lengths of the yarns were 50 mm, which is approximately 85% of the gap between the collectors. The uniformity of the yarn, from SEM imaging along the lengths, suggests good alignment of fibres at the deposition stage.

4. Conclusion

We have been able to produce an array of electrospun fibres that are three-dimensionally suspended between two grounding rings. Lower applied voltages resulted in less fibre splitting and very little interconnectivity. A voltage of 15 kV resulted in straight single electrospun fibres with lengths of between 40 and 100 mm, and a diameter of 1.26 ± 0.19 μm. These suspended fibres can be converted into a multi-filament yarn with diameters below 5 μm by rotating one of the collection rings. The fabrication of such oriented electrospun fibre collections is of significance for tissue engineering of the nervous system, tendons, muscles, or as an artificial extracellular matrix, while the micron-diameter yarns are of interest as sutures for medical applications.

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